



## Metallurgy Department. Progress Report 1983

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# **Metallurgy Department Progress Report for the Period 1 January - 31 December 1983**

Risø-R-503

METALLURGY DEPARTMENT PROGRESS REPORT FOR THE PERIOD  
1 JANUARY TO 31 DECEMBER 1983

Abstract. The activities of the Metallurgy Department at Risø during 1983 are described. The work is presented in three chapters: General Materials Research, Technology and Materials Development, and Fuel Elements. Furthermore, a survey is given of the Department's participation in international collaboration and of its activities within education and training. A list (with abstracts) of publications and lectures by the staff during 1983 is included.

INIS-descriptors: FUEL ELEMENTS, METALLURGY, NONDESTRUCTIVE TESTING, RESEARCH PROGRAMS, RISØE NATIONAL LABORATORY.

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## 1. INTRODUCTION

The time schedule for the establishment of nuclear power in Denmark remains uncertain. Risø is therefore at stand-by in this field but a number of the nuclear programmes are continued in order to ensure up-to-date knowledge. In the Metallurgy Department the nuclear work comprises projects within design and testing of fuel elements, fracture mechanics studies in pressure vessel steels and non-destructive testing. The projects concentrate more and more on advanced problems, which in most cases are studied in collaboration with other parties in Europe and abroad.

With respect to alternative energy technology a major effort is devoted to fibre reinforced plastics. These materials are used for wing-blades for small and medium-size wind turbines built, respectively, by a number of Danish industries and a group of electricity generating companies. Other energy projects include metal-hydrogen systems (e.g. for transportation and storage of energy) and solid ion conductors for use in rechargeable batteries. These projects are partly carried out under the auspices of EEC, The Nordic Fund for Industrial Development and the Ministry of Energy.

Work was done under contract for industries and utilities in Denmark and abroad. Due to their proprietary nature, many of these activities are excluded from the present report. Among the major nuclear activities were fuel element development where collaboration with Atlas Ltd. (former Elsinore Shipyard) was continued, isotope analysis and post-irradiation examinations of full-scale power reactor rods ( $Zr-UO_2$  and  $Zr-UO_2-PuO_2$ ).

Other work on contract was done on high-temperature components for the chemical industry. In 1983 the department started to do work within the European COST-501 project, "High Temperature Materials for Conventional Systems of Energy Generation and

Conversion using Fossil Fuel". In the area of non-destructive testing the work was concentrated on quantitative ultrasonic examinations, especially the characterization of sound fields. Further projects were centered on the development of materials and processes, including ceramics for measurement of oxygen potentials in combustion gases, sintering of ceramics, and brazing technology. In the field of neutron radiography standardization work was continued within the neutron radiography working group sponsored by Euratom.

The department participated in international collaboration on specific research projects and also in a number of international projects and study groups under the auspices of the NEA, EEC and various Nordic and US organizations.

The department organized an OECD-NEA-CSNI/IAEA Specialists' Meeting on "Water Reactor Fuel Safety and Fission Product Release in Off-Normal and Accident Conditions". The meeting was held at Risø 16-20 May 1983, it was attended by about 90 participants, and 35 papers were presented.

The department organized a workshop on "Evaluation of Simulation Techniques for Radiation Damage in the Bulk of Fusion First Wall Materials". The workshop was held at Interlaken, Switzerland, 27-30 June 1983, it was attended by 36 participants and 27 contributions were presented.

The department organized The 4th Risø International Symposium on Metallurgy and Materials Science, 5-9 September 1983. The title of this symposium was "Deformation of Multi-Phase and Particle Containing Materials". The symposium was attended by approximately 100 participants, and 80 papers were presented. Planning was started for the 5th Risø International Symposium to be held at Risø 3-7 September 1984. The title of this symposium is: "Microstructural Characterization of Materials by Non-Microscopical Techniques".

Educational activities were continued; students and graduates from Denmark and abroad studied in the department.



## 2. GENERAL MATERIALS RESEARCH

The materials research programme includes long-term experimental and theoretical studies aimed at contributing to an understanding of the physical mechanisms governing the properties of materials of general technological interest. A large part of this work is carried out in collaboration with universities and research laboratories in Denmark and abroad. Major efforts are devoted to materials preparation and testing and to microstructural characterization by a variety of microscopical and non-microscopical techniques. The relation between the observed macroscopic and microscopic behaviour is studied theoretically in terms of crystal lattice defects by computer methods and combinations of continuum models and discrete models. The research topics include recrystallisation, grain growth and texture evolution (2.1-2.4), high temperature deformation (2.5-2.7), low temperature deformation hardening and fatigue (2.8-2.9), irradiation damage (2.10-2.13), hydrogen in metals (2.14) and solid state ion transport (2.15-2.17). The materials under investigation cover metals and their alloys, composite materials and ceramics. The research results are published in the open literature and for further information the reader is referred to the list of publications, which includes abstracts.

### 2.1. Recrystallization and grain growth in particle containing materials

(In collaboration with the Department of Metallurgy and Materials Science, University of Cambridge)

The grain growth after recrystallization was studied by TEM in aluminium with a dispersion of fine alumina particles. The grain boundary parameters were determined with precision and the pinning force at the particles was determined by analyzing the shape of the pinned boundary. It was found that the pinning force at particles at high angle boundaries is in agreement with

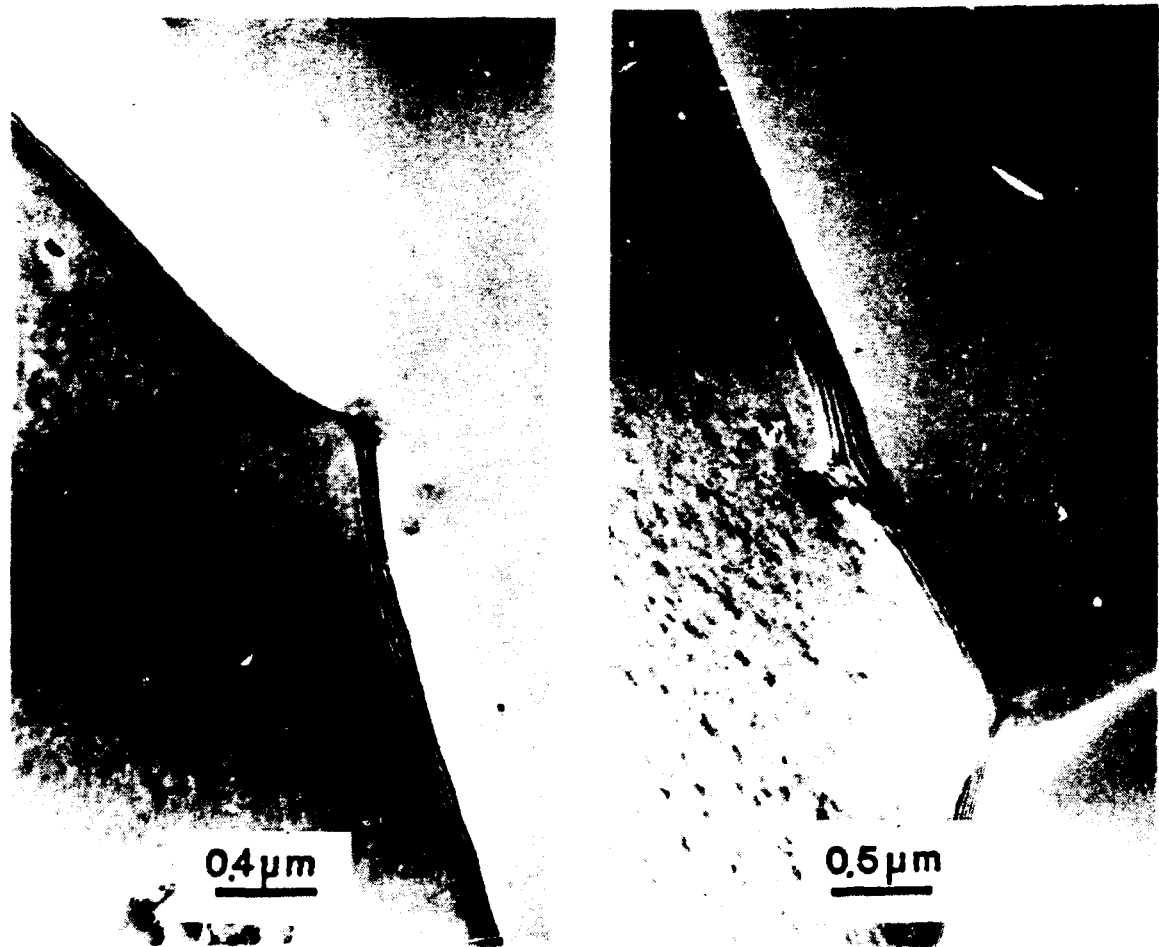


Fig. 1. Typical pinning events during grain growth in aluminium with a dispersion of fine alumina particles. (a) Isolated example of a pinned high angle boundary showing some boundary dislocations in the vicinity of an isolated particle. (b) Pinning interactions in the vicinity of a triple point.

Zener's theory, whereas the pinning force at particles at low angle boundaries is an order of magnitude larger than predicted by Zener. This result is in agreement with earlier observations of pinning at low angle boundaries during subgrain growth in a deformed microstructure.

Work on the migration of high angle boundaries in bi-crystals was extended to cover migration of boundaries in polycrystalline specimens. In-situ measurements were carried out using neutron diffraction techniques and grain size distributions were measured by automatic image analysis.

## 2.2. Neutron diffraction facility for in-situ texture measurements

Improvements were made of the neutron diffraction facility at DR 3 for fast in-situ texture measurements during recrystallization and grain growth. The installation of new motors in the Euler goniometer has reduced the typical recording time for a complete 1/4 pole figure to about 8 minutes. A new furnace system allows samples to be kept in controlled atmospheres, of for example helium, at temperatures of up to about 600°C.

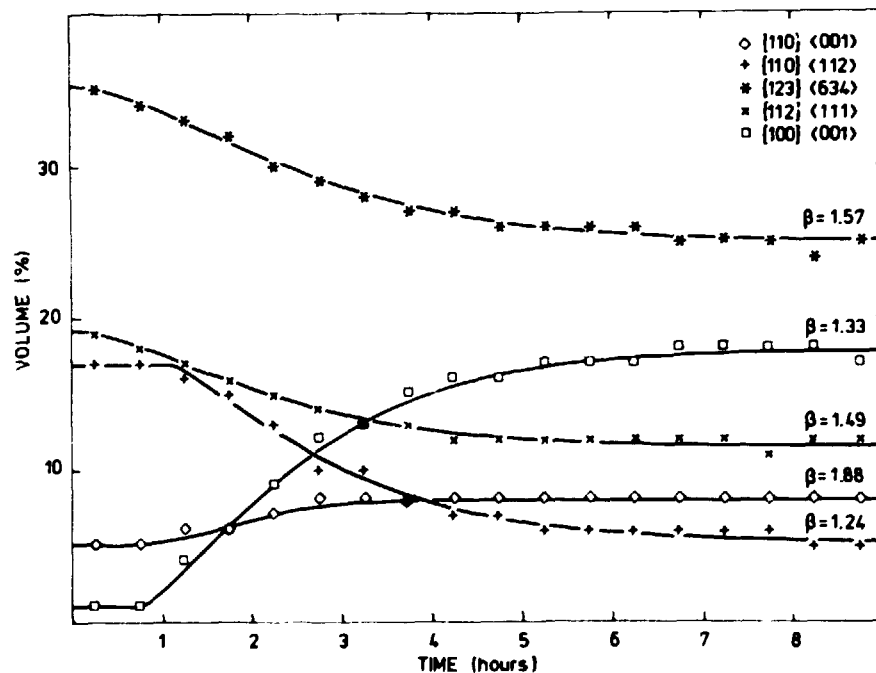


Fig. 2. The development of specific texture components during isothermal annealing at 240°C of aluminium of commercial purity. The volume fractions of each of the components is determined by ODF-calculations based on pole figure measurements.

### 2.3. Texture development in commercially pure aluminium

(In collaboration with the Department of Metallurgy and Materials, Imperial College, London)

The recrystallization of commercially pure (99.4%) aluminium was studied by HVEM and by in-situ texture measurements in the neutron texture goniometer at DR 3. Specimens of 15, 30, 50, 90 and 95% cold rolled aluminium with two initial grain sizes were recrystallized at temperatures between 235 and 330°C while pole figures were measured by neutron diffraction. It was found that the final recrystallized texture in the heavily cold-rolled samples consists of broadened rolling texture components, some random texture and (as for pure aluminium) a cube component whose strength increases with increasing cold-rolling. For the lightly cold-rolled samples no cube component was observed. Texture measurements in local areas in combination with HVEM suggests that the random texture is associated with recrystallized grains, which nucleate at large intermetallic iron-aluminium particles.

### 2.4. Texture development in high-purity copper

(In collaboration with the Department of Metallurgy and Materials Science, University of Cambridge)

Texture changes during normal grain growth in isothermal annealing at temperatures between 450 and 800°C were measured in-situ using the neutron texture goniometer at DR 3. The starting material was cold drawn, recrystallized 99.999% pure copper rods. The  $\langle 111 \rangle$  fibre texture component was found to increase during grain growth, at the expense of the  $\langle 100 \rangle$  component. The ratio  $\langle 111 \rangle / \langle 100 \rangle$  was found to increase with increasing annealing temperature.

### 2.5. Additive strengthening

The basis for addition of strength contributions was reviewed for metals with several mechanisms of strengthening operating simultaneously, such as grain boundaries, particles, forest dislocations, lattice friction. Rules of addition were discussed for two types of obstacles to the plastic deformation: obstacles of intermixed distribution and obstacles of regional distribution. The strength contributions were discussed: the mean stress, the "source-shortening" stress and the forest stress. The rule of addition is the square-root-of-squares for intermixed obstacle distributions; this rule can sometimes be approximated with a linear rule. The rule of addition for regional distributions of obstacles is the volume-weighted average.

The application of these rules to real materials was illustrated. The differences between the resulting strength values from an averaged dislocation arrangement and a localized arrangement were emphasized, and illustrated by the estimates for copper dispersion hardened with silica particles.

### 2.6. Creep of composites

The creep behaviour of fibrous composite materials was analysed on the basis of a macroscopic geometrical model of the composite. The stress contribution, derived from the general two-phase materials were included in the analyses. The creep rate and creep stress are modified relative to those of the (pure) matrix material, and on this basis the creep law for the composite was described. The creep strength is represented by an additive term (friction stress) and a term which is proportional to the matrix stress. The concept of corresponding points on the matrix creep curve (creep rate vs. creep stress) and the composite creep curve suggests the use of a master diagram, which combines data for the creep of the (pure) matrix and the creep of the composite. Several sets of experimental data on metallic composite materials were analysed in terms of the model.

### 2.7. Creep in fcc metals

The recovery of dislocation networks has usually been described by the Friedel model or similar models which predict that the change in average link length per time unit is inversely proportional to the average link length. However, this disagrees with experimental results from the literature on annealing of creep-induced dislocation networks after a stress removal. These results show an initial phase of fast recovery after which the recovery rate decreases rapidly to a value close to zero. A recovery model was therefore developed in which sites for strong recovery are created by the glide process: the intersection of two dislocations of opposite sign creates strongly curved dislocations which will contract under the influence of their line tension and thereby lower the stress needed to break adjacent junctions. The predictions of this model were compared with experimental results from the literature. An example is shown in Fig. 3 where a theoretical recovery curve for Al-11% Zn after a stress removal is compared with experimental results by Hausselt and Blum (Acta Metall. 24 (1976) 1927).

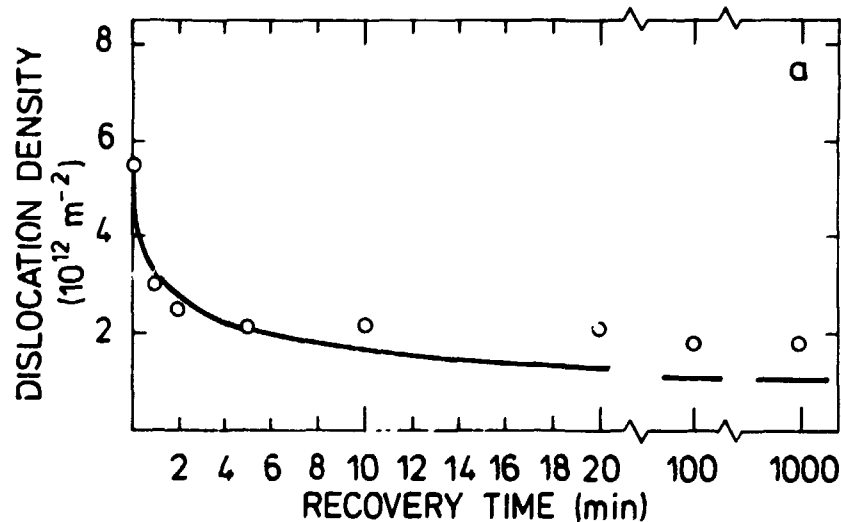


Fig. 3. Theoretical prediction (line) and experimental results from the literature (points) on recovery after a stress removal for Al-11% Zn crept at 523 K under a stress of 1.69 MPa.

The creep properties of particle-containing materials were reviewed together with various models for the effect of particles on basic mechanisms like dislocation creep, diffusional creep and grain-boundary sliding. The strength and shortcomings of the models were considered with a view to suggestions for future research.

### 2.8. Deformation hardening of composites

(Partly in collaboration with the Cavendish Laboratory, Department of Physics, University of Cambridge)

The relationship between continuum models and discrete models for the low temperature strength of plastically heterogeneous crystalline materials was discussed with special reference to Ashby's continuum model and Hirsch's interaction hardening model. Ideas from both models were combined in a new model for work-hardening and slip line spacing in dispersion hardened metals.

In the continuum model it is assumed that the plastic strain can be represented realistically as a uniform inelastic strain. However, in the case of fibre composites with large fibre volume fractions it is not obvious why such a crude picture of plastic flow in the matrix should lead to a realistic theory of deformation hardening. In-situ X-ray diffraction data from the literature were therefore examined in detail. It was found that the concept of an effectively uniform plastic strain in the matrix does in fact give a consistent account of observed mean phase stresses. The experimental data show very directly that the mean matrix stress consists of two terms: an "elastic" term, proportional to the applied stress, and a "plastic" term, proportional to the unrelaxed portion of the plastic strain in the matrix. The X-ray data suggest that the in-situ hardening of the matrix is linear, and that the relaxed portion of the plastic strain in the matrix is proportional to the unrelaxed portion.

A simple model of the Bauschinger effect of a fibre composite emerged after writing the constrained matrix flow stress as a linear combination of the unrelaxed plastic strain and the ap-

plied stress. The model suggests that it is possible to measure the "plastic" matrix hardening separately in a Bauschinger experiment. Preliminary Bauschinger data on copper single crystals with tungsten fibres were analysed in terms of the model.

### 2.9. Fatigue in metals

Recent experimental and theoretical work suggests that the linear theory of composites can be combined with the dislocation theory of obstacle controlled flow to give a unified view of the low temperature micromechanisms of deformation hardening in crystalline matter. A summary of this view was given by using the idea of "hardening diagrams" (or "fatigue diagrams") in which the plastic strain amplitude is taken as one axis and the cycle number as the other axis. Observed boundaries between different regimes of hardening, and observed transitions from one stage of hardening to the next, are drawn in the diagrams. In this way the fields governed by each hardening mechanism are displayed. The hardening diagrams are constructed mainly from measurements of monotonic hardening rates and of the sizes and shapes of stress-strain loops in cyclic hardening at constant plastic strain amplitudes. Observations of the hardening behaviour by different experimenters are fairly consistent, and they have to a large extent been correlated with microstructural observations obtained by microscopical and non-microscopical techniques. At present the most comprehensive data set is for copper single crystals deformed in single slip; but recent work allows a reasonably detailed hardening diagram to be constructed for copper polycrystals. The existing data for the other face-centred cubic metals suggest that their hardening diagrams will not differ qualitatively from those of copper. The existing data are also sufficient for giving a rough outline of the influence of various parameters on the hardening diagrams: crystal orientation, texture, grain-size, particles, solute and temperature. Figure 4 illustrates how the hardening diagrams can be used for making systematic comparisons between different alloys. This



approach was applied in continued experimental studies of the effects of polycrystallinity and hard particles on the fatigue behaviour of copper.

The hardening diagrams give a convenient summary of the successive processes of plastic flow at low temperatures; but they have several limitations: mainly, they are limited to hardening associated with dislocation microstructures which evolve along one particular type of strain path, paths of constant plastic strain amplitudes. The effect of the strain path will therefore be examined in the light of recent theory.

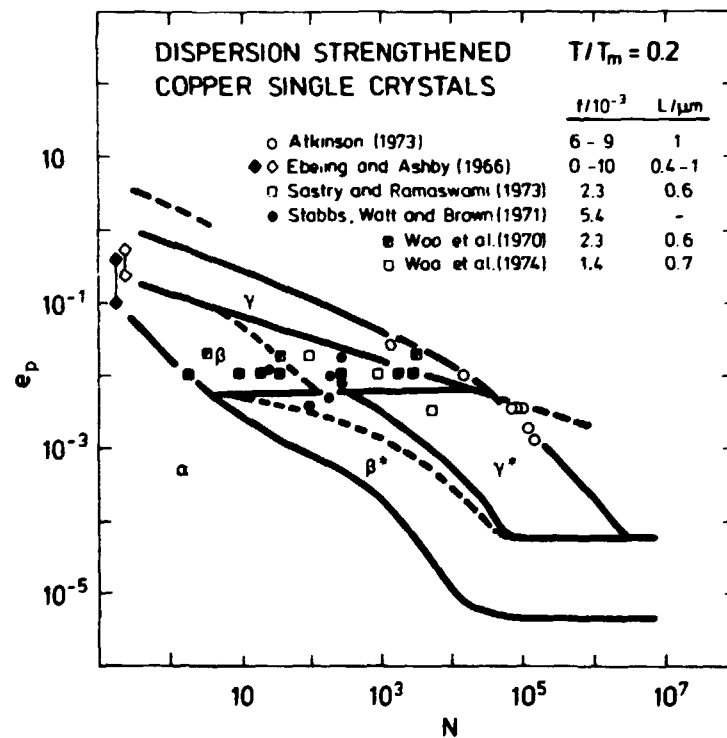


Fig. 4. Data points from the literature for cyclic deformation of dispersion strengthened copper single crystals, plotted on the hardening diagram for pure copper single crystals.

In the continued experimental studies copper polycrystals were prepared with a strong  $\langle 111 \rangle$  fibre texture component by grain growth. The texture was characterised quantitatively by use of the neutron texture goniometer at DR 3. The material was deformed at room temperature in monotonic tension and in tension-

compression at low constant amplitudes of plastic strain. The effect of texture on the monotonic flow stress was reflected in the Hall-Petch relation. In cyclic deformation the  $\langle 111 \rangle$  texture component appears to displace the field for persistent slip in the hardening diagram to lower cycle numbers and to displace the cyclic stress curve to higher stresses.

2.10. Irradiation of high-purity aluminium with 600 MeV protons  
(In collaboration with EIR/SIN, Würenlingen, Switzerland)

Samples of high-purity aluminium (99.999%) were irradiated with 600 MeV protons at the Swiss Institute for Nuclear Research (SIN) to different dose levels (up to 5 dpa) at temperatures in the range of 130-430°C. TEM examination showed that at all irradiation temperatures and displacement doses helium bubbles are



Fig. 5. Helium bubbles at nodal points of a dislocation wall in aluminium irradiated with 600 MeV protons to 2.5 dpa at 116 °C.

formed uniformly through the whole grain interior. The bubble density decreased and the bubble swelling rate increased with increasing irradiation temperature. At all irradiation temperatures bubbles were also observed at grain boundaries: Bubble denuded zones were observed along the boundaries; their width increased with increasing irradiation temperature. At higher irradiation temperatures (320 and 430°C) grain boundaries appeared to have migrated during irradiation. As a result of the migration complicated bubble structures evolve in the zone through which the boundary migrates.

In samples irradiated at temperatures beyond 220°C, precipitates were clearly resolved in the grain interior as well as at the grain boundaries. The precipitates were generally very thin (20-30 nm). The precipitates formed in the grain interior were normally decorated with a large number of helium bubbles.

Bubbles are formed preferentially at matrix-dislocations, at grain boundary dislocations and at grain boundary steps or ledges: An estimate of helium mobility showed that helium atoms diffuse at about the same rate in dislocations as in the lattice. The results (on inter-bubble spacing in the matrix and the width of the bubble denuded zone along grain boundaries) suggest that although the rate of helium generation in these experiments is high, the flux of helium atoms to the boundaries is rather low.

In addition to TEM, Small Angle Neutron Scattering (SANS), Small Angle X-ray Scattering (SAXS) and Positron Annihilation (PA) techniques were used to characterize the damage structure in some of the 600 MeV proton irradiated samples.

### 2.11. Helium diffusion and bubble nucleation

(In collaboration with the Metallurgy Division, AERE Harwell, U.K.)

The problem of helium diffusion during irradiation (i.e. in the presence of vacancies and self-interstitials) was analysed. It was argued that helium atoms can diffuse via the di-vacancy mechanism or the self-interstitial displacement mechanism; it is also possible that both mechanisms operate simultaneously. It was shown that under steady state diffusion conditions, and in the absence of large clusters of vacancies and gas atoms, the effective jump frequency of a helium atom is comparable with that of a single vacancy. This means that in the gas-driven homogeneous nucleation concept, the scale of bubble nucleation should be approximately the same for both diffusion mechanisms. Using the homogeneous nucleation concept where nucleation occurs basically by diffusion and coalescence of mobile vacancy-gas atom clusters, the effect of damage rate and helium generation rate on bubble nucleation was calculated. The bubble density increases as the square root of the damage rate and at a given damage rate the bubble density increases as the square root of the gas generation rate (per dpa). These calculations were carried out for aluminium irradiated with 600 MeV protons at 140°C. They suggest that helium atoms in these experiments are diffusing at a rate which is  $\sim 4 \times 10^{-5}$  times slower than expected (i.e. diffusion coefficient for a single vacancy). The reason for this decrease in helium mobility is not obvious at present.

### 2.12. Irradiation of high-purity aluminium with neutrons

High purity aluminium was irradiated with fast neutrons to a dose of  $5 \times 10^{24} \text{ n} \cdot \text{m}^{-2}$  at 120°C in the DR 3 reactor at Risø. TEM investigation showed that the early stages of void swelling were strongly affected by grain boundaries and dislocation walls. The swelling is enhanced in a peak swelling zone extending up to 10-15  $\mu\text{m}$  into the grain interior of annealed specimens. In polygonized material, which contains dislocation walls delineating a cell-structure, the corresponding effect leads to cell-size de-

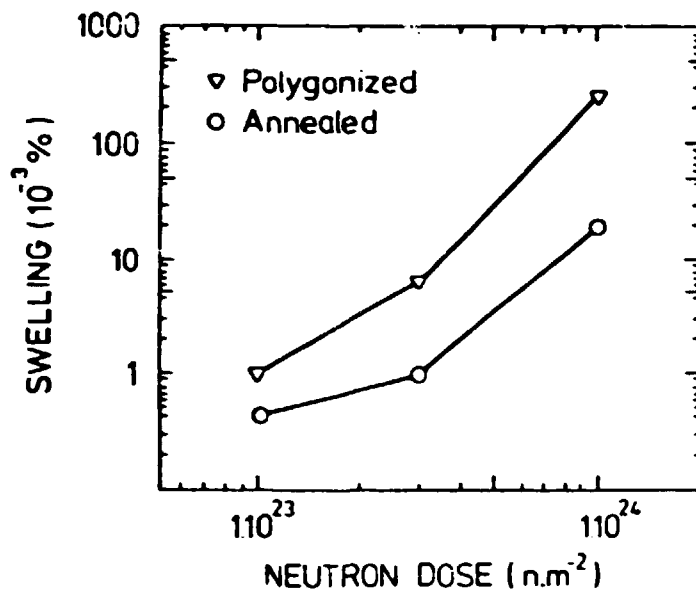


Fig. 6. Effect of microstructure on overall swelling in high purity aluminium irradiated with fast neutrons. The polygonized microstructure, comprising dislocation cell walls, increases swelling by an order of magnitude compared to annealed material.

pendent swelling. A comparison between annealed and polygonized material showed overall swelling to be an order of magnitude higher in the polygonized material.

### 2.13. Computer investigation of radiation damage

A computer analysis of void swelling in materials with heterogeneous distributions of dislocations and voids (so that dislocations and voids are spatially separated) was concluded. The analysis shows that void segregation always leads to reduced swelling rate, the reduction increasing with increasingly coarse segregation. Coarse-scale segregation of the dislocations also leads to reduced swelling rate, whereas fine scale dislocation segregation leads to increased swelling rate. The reduction in swelling for coarse scale segregation is caused by the diffusional barrier between the dislocations and the voids which have to interact to produce swelling. The increase in swelling for fine scale segregation of dislocations is caused by the increased bias of the dislocations at higher dislocation density (which more than balances the diffusional barrier at short distance). The extreme case of dislocation segregation into subgrain boundaries leads to reduced swelling even for segregation on fine scale.

The calculations refer to two experimentally observed examples of materials with heterogeneous distribution of dislocations and voids: Copper irradiated with neutrons to low doses at low dose rates and heavily cold-worked copper irradiated in a high voltage electron microscope. The neutron irradiated copper consists of walls of dislocations (with practically no voids) separating void containing cells (with low dislocation density) in the size range 2-10  $\mu\text{m}$ ; for segregation on this scale the segregation reduces the calculated swelling rate very significantly. In cold-worked copper with a much finer cell structure the segregation increases the calculated swelling rate unless one introduces subgrain boundaries with inter-dislocation spacing of the order of a few nanometers.

#### 2.14. Hydrogen in metals

A study was made of the initial reaction between 99.9999% hydrogen and well-characterized powders consisting of nearly perfect magnesium spheres of narrow size distributions, to which a definite specific surface area was assigned through area measurement by low temperature argon adsorption.

It was found that an annealing procedure, consisting of heating to 400°C for one hour in helium, has a critical effect: Without heating or with heating for a shorter time or at a lower temperature the reaction will not occur or will proceed extremely slowly, stopping at low reaction degrees. It was suggested that the effect of the anneal is to create spots where the hydrogen can penetrate the always present oxide layer. The mode of destruction of the oxide layer seems to depend on its thickness. In thick layers cracking induced by different thermal expansion was observed, whereas thin layers (< 20 nm) of non-crystalline structure may deteriorate by crystallization or solution of oxygen in the magnesium metal.

The initial reaction with hydrogen suggests that the overall reaction rate is determined by surface nucleation in a broad sense. At all degrees of reaction over 0.1 a mixture of pure,

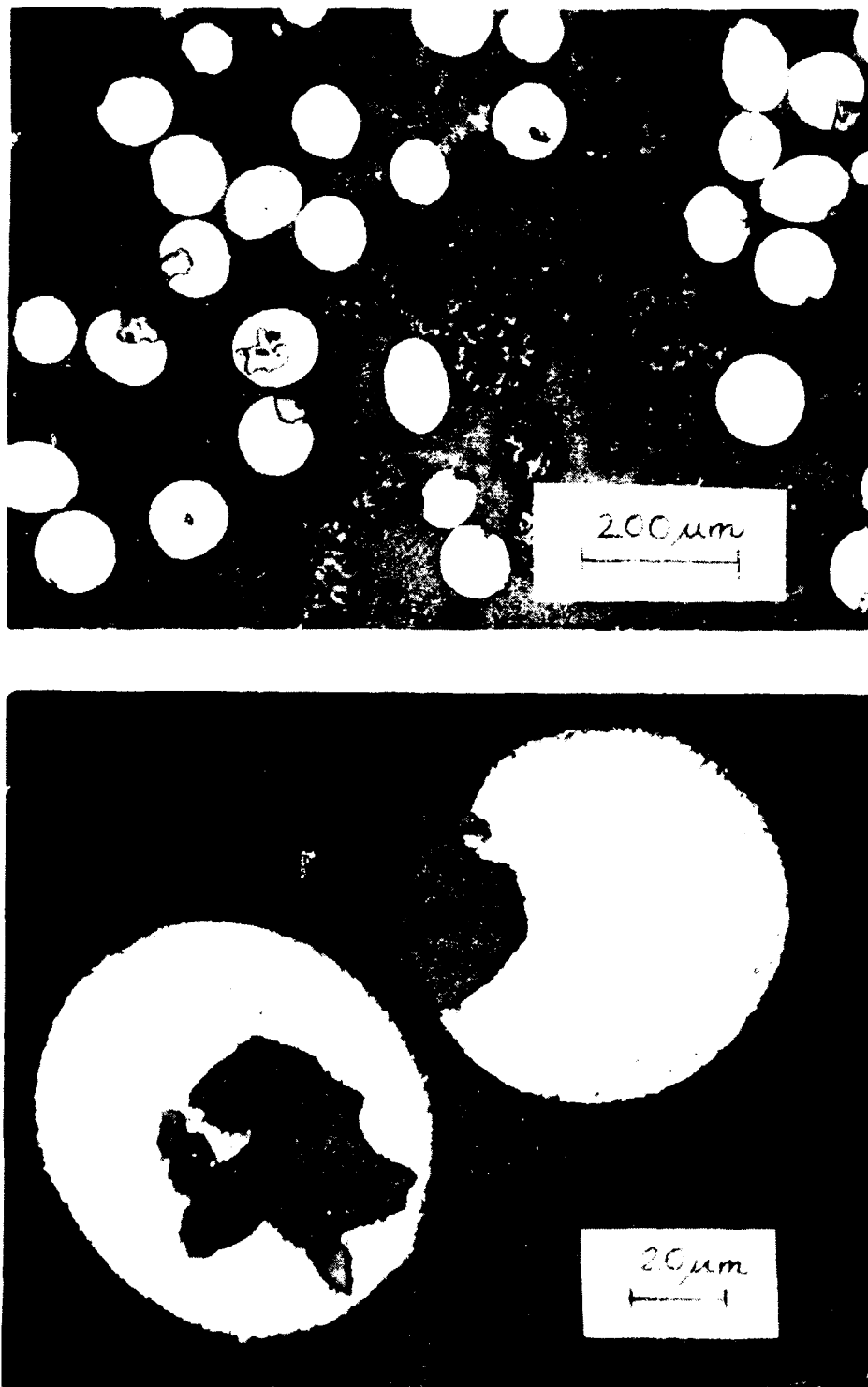


Fig. 7. (a) Magnesium powder exposed to hydrogen, 10% reacted. A few hydride grains (grey) are seen in the magnesium particles (white). The grey-black particles are supporting  $\text{Al}_2\text{O}_3$ . (b) Enlarged view of hydrided magnesium particles.

partly reacted through one nucleus, and completely transformed magnesium particles were found. This shows that the time of transformation of an individual particle, once a nucleus is formed, is short compared to the total reaction time for the entire sample.

### 2.15. New fast ion conductors

(In collaboration with the Physics Department at Risø)

The ionic compounds  $\text{MTaCl}_6$  ( $M = \text{Na}, \text{K}$ ) formed by melting of stoichiometric amounts of  $\text{MCl}$  and  $\text{TaCl}_5$  have proven to form a new interesting group of fast ionic conductors. The transition to the fast ionic phase, which in both cases is observed in the ionic conductivity as a discontinuous increase by two orders of magnitude, occurs at rather low temperature ( $T = 168^\circ\text{C}$  and  $127^\circ\text{C}$ , respectively). The ionic conductivity observed for  $\text{KTaCl}_6$  at  $300^\circ\text{C}$ :  $\sigma = 0.1 \Omega^{-1} \text{cm}^{-1}$ , is the highest value reported for potassium conduction. The discontinuous changes in the ionic conductivities are consistent with the structural studies by neutron powder diffraction which reveal three crystallographic phases of which the high temperature  $\alpha$ -phases both have cubic symmetry. The phase transitions are also evident from specific heat studies but there are significant discrepancies in the transition temperatures compared to the ionic conductivity results.

It was shown that  $\text{NaNbCl}_6$  has similar physical properties and it is expected that mixtures of niobium and tantalum compounds may have lower transition temperatures to the fast ionic phase.



## 2.16. Studies of fluorite type ionic conductors

(In collaboration with the Physics Department at Risø)

Studies of pure and doped ionic conductors with the fluorite type of crystal lattice were continued. The thermodynamic model for fluorites, which has been developed with the purpose of studying the thermal generation of defects from specific heat measurements, was extended to include calculations of the ionic conductivity. With only two adjustable parameters, in addition to those derived from specific heat studies, it was possible to give a satisfactory account for the ionic conductivity data of  $\text{Pb}_{1-x}\text{U}_x\text{F}_{2+2x}$  with  $x = 0, 0.05$  and  $0.10$ .

Diffuse neutron scattering from heavily doped  $\text{Ba}_{1-x}\text{La}_x\text{F}_{2+x}$  ( $x = 0.492$ ) showed evidence for short range correlations between 222-type defect clusters. To investigate these correlations further, the room temperature intensity of the diffuse peaks centered at  $(h \pm \frac{2}{3}, k, l)$ ,  $(h, k \pm \frac{2}{3}, l)$  and  $(h, k, l \pm \frac{2}{3})$  were examined up to  $h^2+k^2+l^2 = 10$  ( $(h, k, l)$  are reciprocal lattice vectors for the fluorite lattice). The 222-cluster is uniaxial in character, and the ionic disorder can be visualized as an assembly of aggregates, where an aggregate consists of aligned close packed 222-type clusters.

## 2.17. Structure of $\text{Li}_x\text{V}_6\text{O}_{13}$

(In collaboration with the Physics Department at Risø, and Fysisk-Kemisk Institut, The Technical University of Denmark)

$\text{Li}_x\text{V}_6\text{O}_{13}$  ( $x \lesssim 8$ ) is a lithium intercalation material with potential applications as cathode material in lithium batteries. The electromotive force relative to lithium metal reveals three flat plateaus which correspond to filling up a total of one (2.76 V), four (2.54 V) and eight (2.16 V) lithiums per  $\text{V}_6\text{O}_{13}$ . This behaviour is usually interpreted to result from three regions composed of two-phase materials. However, it has been shown to be consistent with a simple model which assumes statistical distribution of non-interacting lithiums on three types of energetically different sites in the lattice. Neutron dif-

fraction studies were initiated to study the structural properties of the intercalation process. Diffraction patterns were recorded at room temperature for four different compositions ( $x = 0, 1, 2.1$  and  $7.5$ ) and structural analyses by use of the EDINP-refinement program are in progress. The results from  $V_6O_{13}$  deviate from the structure deduced from X-ray single crystal data (space group  $C2/m$ ), probably due to lack of stoichiometry in the vanadium-oxygen ratio.

### 3. TECHNOLOGY AND MATERIALS DEVELOPMENT

The materials technology programme concentrates on the development of new materials and methods for their fabrication, characterisation and testing. Projects related to alternative energy technology include work on the mechanical properties of fibre reinforced plastics for light and strong components, e.g. wind-mill blades (3.1), work on metal-hydrogen systems for transportation and storage of energy (3.2), and work on materials for battery applications (3.3-3.4). Most of these projects are carried out in collaboration with the industry, partly under the auspices of EEC, the Nordic Fund for Industrial Development and the Ministry of Energy. Work on high temperature corrosion in conventional energy technology (3.6) is carried out within the European COST-501 project. This work is done in collaboration with a Danish firm and partly sponsored by the Ministry of Energy. In the area of non-destructive testing the work is concentrated on quantitative ultrasonic examinations, especially the characterization of sound fields. Further projects are centered on the development of materials and processes, including ceramics for measurement of oxygen potentials in combustion gases (3.5), sintering of nuclear ceramics, and brazing technology (3.7). In the field of neutron radiography standardization work is carried out within the neutron radiography working group sponsored by Euratom. Due to their proprietary nature, some of the projects in the technology programme are excluded from the progress report.

#### 3.1. Fibre reinforced plastics

Research and development on fibre reinforced plastics were continued in the following fields: fabrication technology, testing methods, mechanical properties and design and analysis.

Development of the computer controlled filament winding machine was continued. The software was improved to ensure more accurate placement of the fibre, and a more easy teach-in procedure for new winding patterns on complicated mandrels. The hardware was expanded with an automatic fibre tension controller, and a new wetting apparatus was developed and manufactured. Among other applications the filament winding machine was used to fabricate pressure vessels of fibre reinforced plastics.

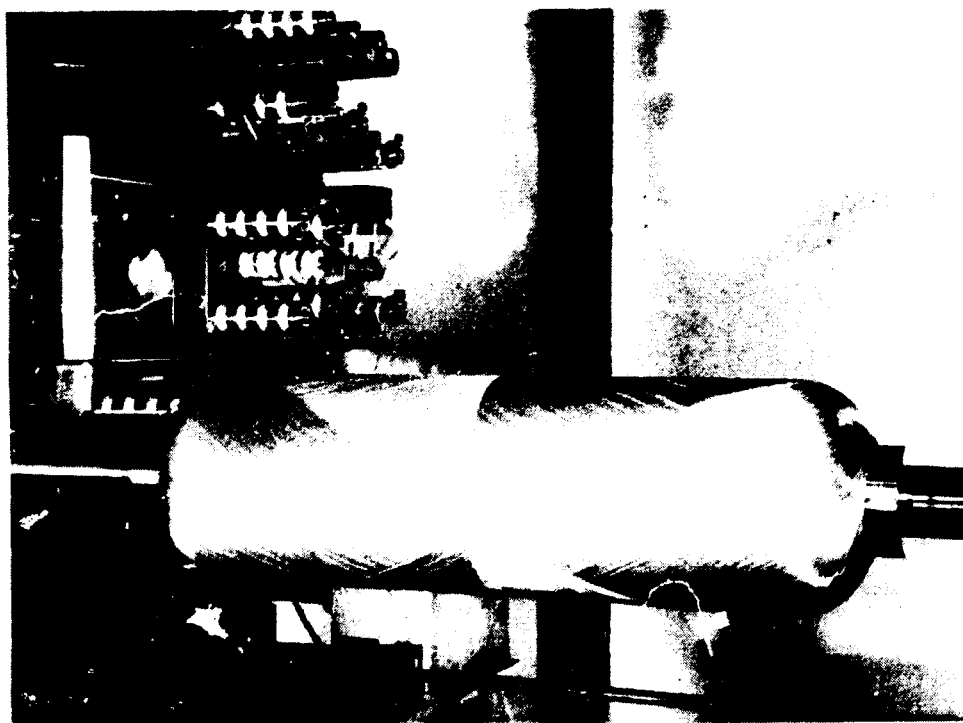


Fig. 8. Fabrication of fibre reinforced pressure vessel by computer controlled filament winding.

Work on damage in angle-ply laminates is carried out to study the influence of fibre orientation, stacking sequence and specimen geometry on the damage development and strength. To study failure mechanisms replicas are taken of the free edge of carbon/epoxy tensile specimens at different strain levels, the edge is examined in an optical microscope and X-ray photography is used together with a contrast liquid to reveal interior cracks or de-

lamination. Scanning electron microscopy is used for examination of the damage zone after ultimate failure. The tensile specimens are cut from plates cured in an autoclave. To ensure that the specimens are without initial damage the plates are examined by ultrasonic C-scan and the edges of the specimens are examined by X-rays and contrast liquid.

### 3.2. Metal-hydrogen systems

Studies of the properties of magnesium powder relevant to storage of hydrogen as magnesium hydride,  $\text{MgH}_2$ , were continued. The influence on the powder of cycling during more than 500 cycles was determined in the case of a pure hydrogen gas (99.9997%) and in the case of hydrogen with 85 ppm oxygen and 8 ppm water vapour. In both experiments a moderate overall reduction in absorbed/desorbed amount of hydrogen was observed with continued cycling. This was ascribed to a reduced absorption rate with increased number of cycling. The effect of oxygen and water vapour was negligible, which was confirmed by precision absorption measurement after the cycling experiment. Despite the decreased absorption rate, mainly at higher degrees of reaction, little change in desorption kinetics was observed.

Experiments were carried out on the safety aspects of handling combustible fine powders. Pure magnesium as well as magnesium hydride powders were ignited and various fire extinguishing techniques were tested. The fires were photographed on infra-red sensitive films. In other experiments water was added to the powders. Only extremely weak reactions were observed (the temperature rose by typically  $20^\circ\text{C}$ ) in consequence of the water addition.

### 3.3. Lithium electrodes for non-reversible batteries

(In collaboration with A/S Helleseus)

Studies were continued on the formation of a passivating LiCl-layer on the lithium anode in non-reversible lithium-thionyl-chloride batteries. Measurements were performed on two types of cells: one in which the lithium metal was in direct contact with an SS container and one in which it was in contact with a glass container. Typical complex plane impedance plots obtained in these measurements are shown in Fig. 9 for two different electrolytes (A and B). The ac-impedance (10 Hz value) determined for the two types of cells is shown in Fig. 10 as a function of time. From this plot it is clear that the rate of passivation is greater in the SS-cells than in the glass cells. Most of the impedance measurements discussed above were performed on a Solartron 1250 Impedance Analyzer and the data were collected automatically by a computer controlled data acquisition system.

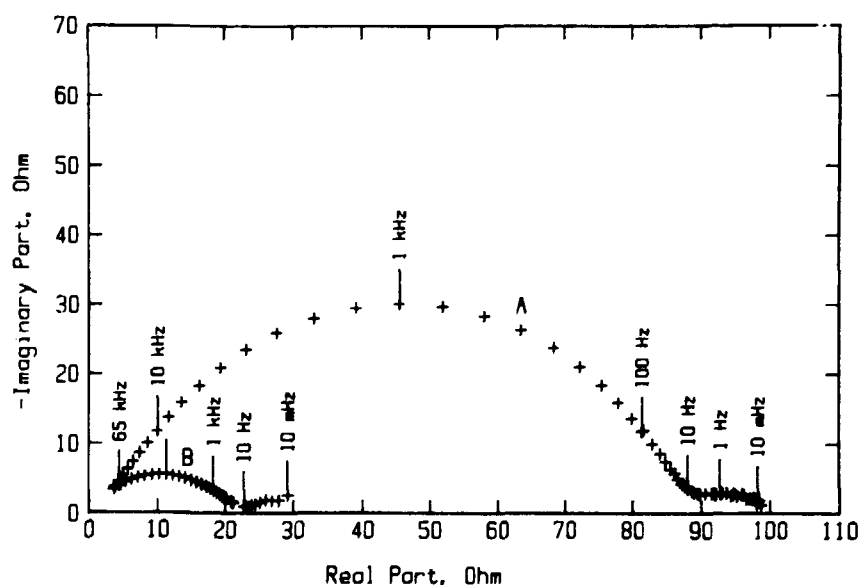


Fig. 9. Complex plane impedance plots measured in studies of the formation of passivating LiCl-layers on lithium anodes in different electrolytes.

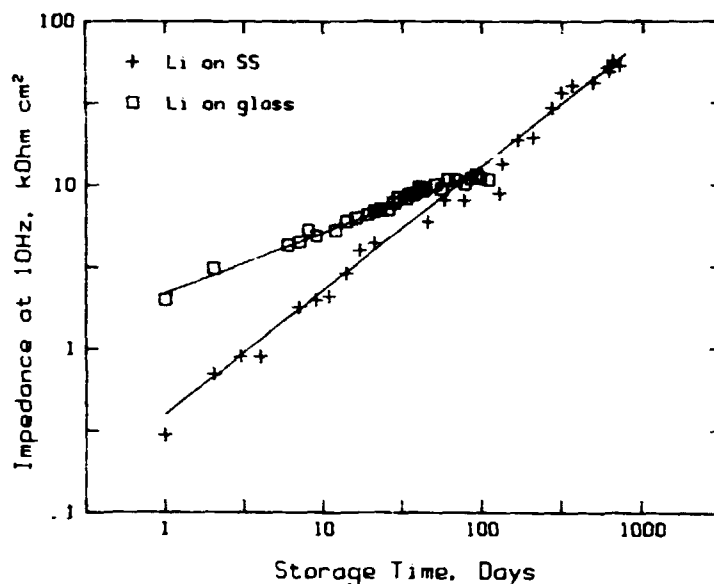


Fig. 10. The ac-impedance for two types of cells measured as functions of storage time.

### 3.4. Lithium-alloy anodes for reversible batteries

(In collaboration with Imperial College, London)

Alloy anodes are expected to present a number of advantages over pure lithium anodes, primarily geometric stability, at a cost of reduced power and energy density. The special type of alloys called intermetallic "compounds" offers a fairly constant voltage until fully charged or discharged, which are indicated by abrupt voltage changes. Binary intermetallic compounds with lithium are formed by the elements of the second, third and fourth main groups and the first and second side groups of the periodic system.

Most of these binary compounds have now been subjected to preliminary investigation. The elements were melted together in a differential thermal analysis (DTA) apparatus; the formation of the intermetallic compound displays itself as an exothermic peak. The compound's ability to accept and release lithium within reasonable voltage limits is subsequently investigated by using the DTA crucible in a cyclic voltammetry experiment.

Transitions between compounds of different stoichiometry are displayed as current peaks. The height of the peaks give indications of the diffusion coefficients for lithium in the compound.

It seems, that the larger (and heavier) the alloying element is, the larger is the diffusion coefficient for lithium and thus the possible current density. In practice, it may be necessary to compromise between the weight of the electrode and its ability to deliver large current densities. The compounds  $\text{Li}_4\text{Sn}$ ,  $\text{LiIn}$ ,  $\text{Li}_2\text{Ca}$ , and  $\text{Li}_3\text{Cd}$  have delivered the largest currents, up to  $2 \text{ mA/cm}^2$ .

At least some of the alloy electrodes can be produced as large-area foil by electroplating lithium on a sheet of the alloying element. The formation of the intermetallic compound occurs during plating. This was thoroughly investigated with aluminium. Total conversion leads to disintegration, but the unconverted part of the sheet eliminates the otherwise necessary current collector. Electrodes containing  $15 \text{ C/cm}^2$  on a  $100 \text{ }\mu\text{m}$  thick Al sheet were produced routinely. Such an electrode can be cycled with good efficiency up to some  $\text{mA/cm}^2$ .

### 3.5. Oxygen conductors

Oxygen conducting oxides with a high conductivity are of interest as electrolytes for oxygen sensors and fuel cells. Hitherto these systems were based on doped zirconia, which, however, require a high operating temperature due to a relatively low conductivity. With the aim of developing new oxygen conductors with an improved conductivity, which would reduce the required operating temperature, the ceria-gadolinia system was examined.

The methods developed for fabrication of test specimens of these oxides comprise: Homogeneous coprecipitation of carbonates (by hydrolysis of urea), calcination in air, ball milling, isostatic pressing (discs and tubes) and sintering. The methods used to



characterize the specimens were: X-ray (structure), dc- and ac conductivity measurements, Scanning Electron Microscopy (Gd-distribution) and Thermogravimetry (chemical and thermal stability).

It was found that high density specimens with a homogeneous distribution of Gd and with very good chemical and thermal stability can be obtained for any composition in the ceria-gadolinia system. The highest conductivities of the specimens examined was obtained for the composition  $\text{CeO}_2/4 \text{ mole } \% \text{ Gd}_2\text{O}_3$  ( $\text{Ce}_{0.92} \text{Gd}_{0.08}\text{O}_{1.96}$ ). Typical  $\log \sigma - 1/T$  plots (Arrhenius plots) give the curves both for the grain boundary and lattice conductivities. A change of slope observed in the curve for the lattice conductivity is attributed to a change in the conduction mechanism due to a change in the defect structure from fully associated oxygen vacancies (low temperature) to free oxygen vacancies (high temperature). Finally the X-ray structures observed for sintered specimens were for  $(\text{Ce}_{1-x} \text{Gd}_x)\text{O}_{2-x/2}$ :  $0 \leq x \leq 0.4$ -fcc;  $0.4 < x \leq 0.75$  - two-phase region of fcc and bcc;  $0.75 < x \leq 0.95$  - bcc;  $0.95 < x \leq 1$  - two-phase region of bcc and monoclinic.

### 3.6. High temperature corrosion

The phenomenon of metal dusting was studied. Metal dusting is a form of deterioration that occurs with iron-, nickel-, and cobalt-based alloys in gases containing carbon monoxide and/or hydrocarbons at temperatures in the approximate range of 400-900°C. The active reaction is considered to be accelerated carburization, frequently producing pits containing a powder of corrosion products: carbides, oxides, unreacted metal particles, and graphite. Samples from an industrial case of metal dusting were examined by electron microscopy. The still solid, but severely carburized surface layer, shows precipitation of chromium-enriched particles of typical dimensions less than 1  $\mu\text{m}$ .

In collaboration with A/S Haldor Topsøe, an investigation of the conditions for the occurrence of metal dusting was started.

Specimens will be exposed to gases of well-defined oxygen and carbon activities. With this project, we participate in the inter-european high-temperature materials collaboration COST 501.

### 3.7. Brazing and soldering

Investigations of vacuum-brazed joints in high temperature materials were continued. Tests were made of the corrosion resistance in 1-3%  $\text{HNO}_3$  of vacuum-brazed joints in an 18/8 stainless steel. It was found that the corrosion process depends on the chromium content of the brazing alloy and on the amount of certain intermetallics.

Contract work was continued on industrial applications of dip-brazing, vacuum brazing and ultrasonic soldering of aluminium as well as vacuum brazing of stainless steels, aluminium alloys and nickel alloys.

#### 4. FUEL ELEMENTS

The Danish water reactor fuels programme continues to utilize the irradiation facilities in the DR 3 materials testing reactor at Risø and the OECD Halden Reactor in Norway. Extensive post-irradiation examinations are performed in the Risø hot cells. An internationally sponsored, three-year project, "The Risø Transient Fission Gas Release Project" is now well underway. This is a follow-up program to the "Risø Fission Gas Project" that was executed in 1980-81. Additional information on fuel performance becomes available from international collaboration arrangements, i.e. the OECD Halden Reactor Project (Norway), the "Super-Ramp" and "TransRamp" projects at Studsvik (Sweden) and Battelle's "High Burnup Effects Program" (USA). Outside these national and international programmes, valuable experience is gained from examination in the Risø hotcells, under special contracts, of foreign water reactor fuels. An important part of the current work comprises Pu-enriched fuel irradiated in the Italian Garigliano reactor up to 25,000 MWD/t.

##### 4.1. UO<sub>2</sub>-Zr irradiations

Standard fuel pins currently under irradiation in the DR 3 reactor have reached maximum burnup levels of 61,000 and 63,000 MWD/tU for BWR and PWR type fuel respectively. The irradiation facilities are also used for testing of LOWI duplex fuel (max. burnup 39,000 MWD/t, see also previous progress reports) and for transient tests with high-burnup fuel in the international fission gas project (see below).

The three Danish test fuel elements in the Halden reactor have now reached the following estimated burnups (average assembly, after correction for depletion):

IFA No.	165	201	202
MWD/tU	42,000	41,000	37,000

#### 4.2. The Risø transient fission gas release project

Late in 1982, a new international project was started, with the objective to study the kinetics of fission gas release in high-burnup fuel. The project period is three years and the sponsors are fuel suppliers, utility organizations, safety authorities and research organizations from Europe, United States and Japan.

This project utilizes fuel partly from one of the Danish Halden assemblies and partly from a commercial BWR power reactor. The fuel is refabricated in the hotcells and mounted with pressure transducers. During the subsequent transient testing in DR 3, this change in internal pin pressure is monitored continuously. Before and after the transient testing, the fuel is characterized extensively in the hot cells.

Important test parameters in this project are: Transient power (up to 450-500 W/cm), burnup (13,000-50,000 MWD/tU) and fill gas (Xe, He at various pressures).

#### 4.3. Retained gas measurement

Local measurement of the content of retained fission gas is an important part of the fission gas studies, especially for fuel pins with a pronounced axial power shape.

A method has been developed for the analysis of pellet-size samples of irradiated fuel. The sample material is oxidized in molten  $\text{NaNO}_3$  containing about 10%  $\text{NaOH}$  and 5%  $\text{Na}_2\text{O}_2$ . During this oxidation the fission gases are released. The gas volume is measured and the gas is analyzed by mass spectrometry.

Figure 11 shows an example of the results obtained in the previous Risø fission gas project. At a burnup of 33,000 MWD/tU, the fuel pin was transient tested in a flux gradient with a maximum around the 250 mm elevation, corresponding to a local heat load of 424 W/cm for 24 h. Such data can then be used for

calculation of axially local fission gas release as a function of local heat load, for verification of performance code calculations.

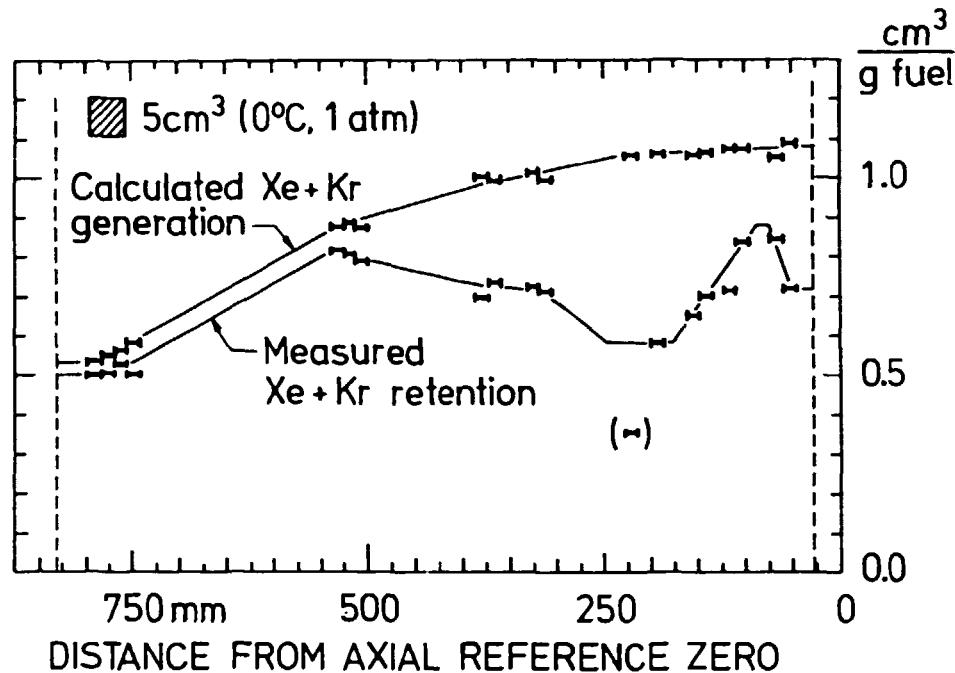


Fig. 11. Calculated gas generation and measured gas retention profiles for a pin tested in the Risø Fission Gas Project.

#### 4.4. Code comparison

As part of an IAEA program, results from calculations with 16 foreign fuel performance codes have been compared. The basis for the exercise was a set of experimental data from the Danish test fuel irradiations, with emphasis on transient fission gas release at high burnup. Design and irradiation data were circulated to the participants. After submission of the calculation results, the observations from hotcell examinations were distributed.

Most of the codes were in reasonable agreement with the experimentally estimated base irradiation release. Only a few of the codes predicted the majority of the release to occur during the

transient testing which the experiment indicated. More basic knowledge seems to be required regarding fast (gaseous) swelling in transients (including the influence of burnup), fast transient gas release, and grain growth at high burnup.

#### 4.5. Medium Enriched Uranium (MEU) fuel for research reactors

The general trend to switch to lower uranium enrichment for research reactor fuel has prompted the development of MEU fuel for the DR 3 reactor. These elements are of essentially the same design as is used for elements of highly enriched uranium, except that the uranium density in the core alloy was increased from 0.6 to 1.2 gU/cm<sup>3</sup>. Irradiation of such fuel element showed that the mechanical integrity remained unchanged, a 1-2% lower thermal flux and a slight increase in fast flux.

## 5. PARTICIPATION IN INTERNATIONAL COLLABORATION

The department is engaged in the following types of international collaboration: joint technical projects, committee work, reception of research fellows, and technical and scientific meetings.

The department was represented on the following committees:

The Information Exchange Group under the European Space Agency on Carbon Fibre Reinforced Plastics,

The Halden Programme Group,

The IAEA International Working Groups on "Reliability of Reactor Pressure Components" and "Water Reactor Fuel Performance and Technology",

The Super-Ramp and Trans-Ramp Project Committees, The Project Committee of the Batelle High Burnup Performance Programme (HBEP), The Principal Working Group No. 3 (Primary Circuit Integrity) of the NEA Committee on the Safety of Nuclear Installations (CSNI),

The COST 501 Committee on Materials for Energy Conversion using Fossile Fuels,

The EEC Advisory Committees for Programme Management: "Plutonium and Transuranium Elements" and "High Temperature Materials",

The European Coal and Steel Community, Executive Committee No. 5: Failure Mechanisms and Design,

The Euratom Neutron Radiography Working Group,

The Council of the International Confederation of Thermal Analysis,

The Nordic Committee for Thermal Analysis,

The Technical Commission of the International Institute of Welding, Commission I, "Gas Welding and Allied Processes", Subcommission A, "Brazing and Surfacing",

Working Party for "Advanced Materials-Transport" of the CREST subcommittee for Raw Materials,

and The EEC Advisory Committees for the implementation and planning of the NET/Technology Programme: "Structural Materials" and "Breeding Materials".



## 6. EDUCATION AND TRAINING

N. Hansen and K. Rørbo gave regular lectures on materials science to students at the Danish Academy of Engineering. C.P. Debel, Aa. Lystrup, O. Bøcker Pedersen, B.N. Singh and O. Toft Sørensen lectured on physical materials science to students at the Technical University of Denmark. N. Hansen, T. Leffers and H. Lilholt acted as external examiners at examinations for the Technical University of Denmark, and O. Toft Sørensen acted as external examiner at examinations for the Technical University of Norway, Trondheim.

### Post-graduate projects

One post-graduate student from the Technical University of Denmark worked in the department on the following project in preparation for his licentiate (Ph.D.) thesis:

E.E. Salah Soliman	Grain Growth of Uranium Dioxide During Irradiation.
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### Degrees conferred

The Technical University of Denmark conferred the degree of lic.techn. (Ph.D.) on D. Juul Jensen.

## PUBLICATIONS

### Deformation of Multi-Phase and Particle Containing Materials.

J.B. Bilde-Sørensen, N. Hansen, A. Horsewell, T. Leffers and H. Lilholt (editors). Proceedings of the 4th Risø International Symposium on Metallurgy and Materials Science, Risø, 5-9 September 1983. (Risø National Laboratory, Roskilde, 1983) 596 pp.

The proceedings contain 9 invited papers and 71 contributed papers. Most of these papers focus on the relation between the mechanical properties and the microstructural features - including the changes in dislocation structure and the possible changes in phase/particle morphology during deformation.

### Metallurgy Department Progress Report for the Period 1 January to 31 December 1983.

Risø-R-486 (1983) 62 pp.

The activities of the Metallurgy Department at Risø during 1982 are described. The work is presented in three chapters: General Materials Research, Technology and Materials Development, Fuel Elements. Furthermore, a survey is given of the department's participation in international collaboration and of its activities within education and training. A list (with abstracts) of publications and lectures by the staff during 1982 is included.

### Creep in Particle-Containing Materials.

J.B. Bilde-Sørensen, In: Deformation of Multi-Phase and Particle Containing Materials. Proceedings of the 4th Risø International Symposium on Metallurgy and Materials Science, Risø, 5-9 September 1983. Edited by J.B. Bilde-Sørensen, N. Hansen, A. Horsewell, T. Leffers and H. Lilholt (Risø National Laboratory, Roskilde, 1983) 1-14.

The presence of particles in a material may result in a considerable increase in the high-temperature strength of the material. Various detailed models have been suggested in the literature for the effect of particles on basic mechanisms like dislocation creep, diffusional creep and grain-boundary sliding, and major current ideas in the field are summarized in this review.

In particular, the occurrence of threshold stresses for the deformation processes and the possible origins of these threshold stresses are considered. The strength and shortcomings of the various models are discussed with a view to suggestions for future research.

#### Scanning Transmissionselektronmikroskopi og Relaterede Analysemetoder (Scanning Transmission Electron Microscopy and Related Analytical Techniques).

J.B. Bilde-Sørensen, In: Metallurgens Værktøj II (The Metallurgist's Tools II). Dansk Metallurgisk Selskabs Vintermøde, Kolding, 5-7 Januar 1983. Edited by E.W. Langer and G. Skjelsager (Dansk Metallurgisk Selskab, Lyngby, 1983) 189-201.

The principles of scanning transmission electron microscopy (STEM) are described. Related analytical techniques like microdiffraction, energy dispersive X-ray spectrometry (EDS) and electron energy loss spectrometry (EELS) are described and the range of their applicability is discussed.

#### Moderne Udmattelsesprøvning. (Modern Fatigue Testing).

P. Brøndsted, In: Metallurgens Værktøj II (The Metallurgist's Tools II). Dansk Metallurgisk Selskabs Vintermøde, Kolding, 5-7 Januar 1983. Edited by E.W. Langer and G. Skjelsager (Dansk Metallurgisk Selskab, Lyngby, 1983) 265-278.

The use of servocontrolled mechanical testing machines in studies of cyclic deformation and fracture was described.

#### Kvantitativ Metallografi - Billedanalyse. (Quantitative Metallography - Image Analysis).

P. Brøndsted, In: Metallurgens Værktøj II (The Metallurgist's Tools II). Dansk Metallurgisk Selskabs Vintermøde, Kolding, 5-7 Januar 1983. Edited by E.W. Langer and G. Skjelsager (Dansk Metallurgisk Selskab, Lyngby, 1983) 63-74.

The manual, semi-automatic and automatic methods whereby two-dimensional patterns may be characterised quantitatively were described. The principles of stereology were outlined.

Cyclic Deformation of Copper and Copper-Alumina Polycrystals.

P. Brøndsted, J.B. Bilde-Sørensen and O.B. Pedersen,

In: Deformation of Multi-Phase and Particle Containing Materials. Proceedings of the 4th Risø International Symposium on Metallurgy and Materials Science, Risø, 5-9 September 1983. Edited by J.B. Bilde-Sørensen, N. Hansen, A. Horsewell, T. Leffers and H. Lilholt (Risø National Laboratory, Roskilde, 1983) 179-188.

Preliminary results are described of a study of the combined effects of polycrystallinity and non-shearable  $\text{Al}_2\text{O}_3$  particles on the fatigue of Cu at constant plastic strain amplitudes. The rapid hardening stage was found to be strongly affected both by polycrystallinity and by the particles. Electron microscopy shows that PSBs do not control the cyclic deformation of fine-grained Cu and Cu- $\text{Al}_2\text{O}_3$  at a tensile plastic strain amplitude of  $10^{-3}$ . The dislocation microstructures in saturated Cu and Cu- $\text{Al}_2\text{O}_3$  are modified by the grain-boundaries, but they are only weakly affected by the particles.

Characteristics of Brazed Joints in High Temperature Materials.

J. Christensen and G.E.K Sheward, In: Proceedings of a Conference on the Behaviour of Joints in High Temperature Materials, Petten, 14-15 May 1981. Edited by T.G. Gooch et al. (Applied Science, London, 1982) (EUR 8021) 117-164.

The abstract appeared in the progress report for 1981 on p. 65.

Characteristics of Brazed Joints in High Temperature Materials.

G.E. Sheward and J. Christensen, presented at the 4th International Conference held by the British Association for Brazing and Soldering, London, 1-3 November 1983. (Available as UKAEA Report No. DCO 7568(S)).

Brazed joints are being increasingly used to manufacture assemblies for high temperature applications - in many cases for use in critical situations in which the penalty of failure is high. Although much information has been published on the properties of brazed joints, the data is frequently related to a specific application, and so there is a shortage of general information on the basic properties of brazed joints made in high temperature materials. This paper will therefore review their characteristics, discussing the effects of the brazing cycle, the parent metal/filler metal reactions, component and joint design and heat treatment on the properties of the brazed component. The

application of quality control procedures can be applied with confidence and make the high temperature brazing process one of the most reproducible of all the joining processes. Finally, recommendations are made about further research and development to obtain a better knowledge of the basic nature brazed joints and the brazing process, so that it can be more widely applied with greater confidence.

Kærvsehedsprøvning i Sammenligning mellem Slagseheds- og CTOD-Prøvning (Measurements of Notch Toughness: a Comparison between Impact Testing and CTOD Testing).

C.P. Debel, In: Metallurgens Værktøj II (The Metallurgist's Tools II). Dansk Metallurgisk Selskabs Vintermøde, Kolding, 5-7 Januar 1983. Edited by E.W. Langer and G. Skjelsager (Dansk Metallurgisk Selskab, Lyngby, 1983) 281-292.

Two test procedures for measuring the notch toughness of metallic materials are considered and compared. One is the older method of impact testing of Charpy-V-Notched bend specimens in order to obtain a toughness transition curve; the other method is the fracture mechanics based Crack Tip Opening Displacement (CTOD) method.

Experience with Ductile Crack Growth Measurements applying the DC-PD Technique to Compact Tension Fracture Specimens.

C.P. Debel and F. Adrian, In: Proceedings of a CSNI Workshop on Ductile Fracture Test Methods, Paris, 1-3 December 1982 (Nuclear Energy Agency, OECD, Paris, 1983) 181-194.

The abstract appeared in the previous progress report p. 47.

Applications of Fluorometallic Screens for Paper Radiography.

J.C. Domanus, Risø-M-2395 (1983) 29 pp.

After the description of the fluorometallic screens and their spectral sensitivity their sensitometric properties are reviewed. Characteristic curves and exposure charts were computed for the structurix IC paper, exposed with ordinary fluorescent IC II as well as fluorometallic RCF screens. From the relative speed, contrast and exposure latitude were computed. Radiographic image quality was investigated using ISO wire IQI's and ASTM penetrometers and the constant exposure methods. The investigation has shown that it is possible and advantageous to use fluorometallic screens for paper radiography, especially above the low kilovoltage range.

### Special Equipment for Etching Nitrocellulose Film.

J.C. Domanus, Risø-M-2396 (1983) 29 pp.

Nitrocellulose film and converter screens used for neutron radiography are described. Difficulties in visualization of radiographs on those films are mentioned. Because there is no equipment for etching nitrocellulose film available on the market Risø has designed and produced such equipment at an estimated cost of Dkr. 15,000. Design criteria for this equipment are given and its performance described.

### Constant Exposure Technique in Industrial Radiography.

J.C. Domanus, Risø-M-2398 (1983) 30 pp.

The principles and advantages of the constant exposure technique are explained. Choice of exposure factors is analyzed. Film, paper and intensifying screens used throughout the investigation and film and paper processing are described. Exposure technique and the use of image quality indicators are given. Methods of determining of radiographic image quality are presented. Conclusions about the use of constant exposure vs. constant kilovoltage technique are formulated.

### Standardization Activities of the Euratom Neutron Radiography Working Group.

J.C. Domanus and A. Laparte, In: Proceedings of the 10th World Conference on Non-Destructive Testing, Moscow, 20-28 August 1982. 243-252.

Activities of the Euratom Neutron Radiography Working Group are revised. Classification of defects revealed by neutron radiography is illustrated in a special atlas. Beam purity and sensitivity indicators are tested together with a special calibration fuel pin.

### Ultralyd Scanning viser skjulte fejl (Ultrasound Scanning reveals Defects).

H.E. Gundtoft, Svejsning 10, No. 1 (1983) 25-28.

At Risø National Laboratory we have worked with the problems of finding and documenting the internal structure and defects in materials and components. This article presents our work with ultrasonic equipment and scanning apparatus developed by us. A minicomputer is used for controlling the inspection

as well as for evaluation and presentation of the results. Results from three different areas of our work are presented.

- Hydraulic scanning system
- Rotating Sound Field scanning examination
- Automatic Tube examination system.

#### Automatic Quantitative Non-Destructive Examination in Computerized Scanning Systems.

H.E. Gundtoft, In: Proceedings of the Automatic Non-Destructive Testing Topical Seminar, University of Idaho, Idaho Falls, 28-30 June 1983 (Southern Idaho Section ASNT, 1983) 103-113. (Also in Risø-M-2391 (1983) 10 pp.).

By some types of nondestructive examination the (small) variation in internal structure is explored, and hence it is important to possess a stable scanning system. Therefore, we have designed and built such a scanning system which is controlled by a computer.

To obtain a fast scanning speed, the x- and y-movements are made by hydraulic cylinders and the position is measured by glass rulers. A PDP11 minicomputer is used to control the movement and to store the measurements together with the position in the computer memory, the z-movement and the tilting movement are controlled by stepmotors. The materials are examined for ultrasonic velocity and damping, and furthermore the dimension (and hereby also the lamination) can be measured.

#### Risø 25 År (Risø 25 Years).

N. Hansen, Berlingske Tidendes Kronik, 9 Juni 1983.

A review of the (political) history of Research Establishment Risø during a 25-years period starting with the foundation in the mid fifties.

#### Nekrolog over Super Sara (Obituary on Super Sara).

N. Hansen, Berlingske Tidendes Kronik, 20 September 1983.

The EEC safety experiment Super Sara was stopped in 1983. The reasons for the ill fate of this project are analysed.

## The Dependence of Flow Stress upon Grain Size for Non-Ferrous Metals and Alloys.

N. Hansen, In: Yield, Flow and Fracture in Polycrystals, Proceedings, Glasgow, 15-16 September 1982. Edited by T.N. Baker (Applied Science, London, 1983) 311-350.

The abstract appeared in the previous progress report p. 50.

## Additive Strengthening in Copper-Alumina Alloys.

N. Hansen and B. Ralph, In: Deformation of Multi-Phase and Particle Containing Materials. Proceedings of the 4th Risø International Symposium on Metallurgy and Materials Science, Risø, 5-9 September 1983. Edited by J.B. Bilde-Sørensen, N. Hansen, A. Horsewell, T. Leffers and H. Lilholt (Risø National Laboratory, Roskilde, 1983) 285-293.

Tensile tests have been performed on polycrystalline samples of copper containing different dispersions of alumina particles. Above a tensile strain of 0.05, the true stress/true strain curves are seen to be parallel, the flow stresses rising with an increase in the alumina particle density. An analysis of this data is given in terms of the additivity of strength components arising from grain boundaries, particles and matrix. In turn, a dislocation interpretation of these contributions is given.

Some of the tensile tests have been interrupted to provide samples for microstructural analysis by transmission electron microscopy (after true strains of 0.05, 0.10 and 0.20). Micrographic evidence in support of the additivity interpretation is provided. In particular, it is observed that the particles contribute to both the strength of the intragranular regions and to the volumes associated with the boundaries.

## Cold-Rolling and Recrystallization of Commercial Purity Aluminium.

B. Bay and N. Hansen, In: The Metallurgy of Light Alloys, Spring Residential Conference, Loughborough, 24-26 March 1983. (The Institution of Metallurgists, London, 1983) (Spring Residential Conference No. 20) 231-235.

In aluminium of commercial purity (99.4%) the recrystallization behaviour was studied as a function of parameters such as the initial grain size (370 and 19  $\mu\text{m}$ ) and the degree of deformation by cold-rolling (5 to 90%). The formation of nuclei was followed by light microscopy and transmission electron



microscopy, and it was observed that the effectivity of nucleation sites such as grain boundaries and deformation bands was enhanced by the  $\text{FeAl}_3$  particles present in the aluminium. The nucleation temperature, the recrystallization temperature and the recrystallized grain size were measured and related to the microstructural observations.

### The Influence of Small Particles and Grain Boundaries on the Deformation Structure of Aluminium.

B. Bay and N. Hansen, In: Deformation of Multi-Phase and Particle Containing Materials. Proceedings of the 4th Risø International Symposium on Metallurgy and Materials Science, Risø, 5-9 September 1983. Edited by J.B. Bilde-Sørensen, N. Hansen, A. Horsewell, T. Leffers and H. Lilholt (Risø National Laboratory, Roskilde, 1983) 145-152.

The deformation structure of samples of commercial purity aluminium and similar samples containing a fine dispersion of small  $\text{Al}_2\text{O}_3$  particles was examined by transmission electron microscopy supplemented with observations of slip line patterns on the surface. The samples were produced in coarse-grained and fine-grained states and cold-rolled to 5% and 15% reduction in thickness.

The deformation structure in the specimens deformed 5% was strongly affected by the presence of the  $\text{Al}_2\text{O}_3$  particles, whereas the effect of particles was much less pronounced in the specimens cold-rolled to 15%. The effect of grain boundaries on the deformation structure was relatively small in all the samples. The deformation structures examined are discussed and related to the flow stress of the specimens determined by tensile testing.

### Grain Growth in Samples of Aluminium Containing Alumina Particles.

C.J. Tweed, N. Hansen and B. Ralph, Metall. Trans. 14 A (1983) 2235-2243.

A study of the two-dimensional and three-dimensional grain size distributions before and after grain growth treatments has been made in samples having a range of oxide contents. In order to collect statistically useful amounts of data, an automatic image analyzer was used and the resulting data were subjected to a series of statistical tests which evaluate the difference between related distributions. Normal grain growth was observed in samples of the lowest oxide content; as the oxide level was increased, a significant narrowing of the grain size distribution was observed. In the latter samples the maximum or limiting grain size measured after prolonged heat treatment is approximately one half that predicted by Zener and is in broad agreement with the values calculated from the models due to Hillert and Gladman. Anomalous grain growth has been observed in samples where normal grain growth is restricted and where the initial grain size distribution is very wide.

### Conductivity, Structure and Specific Heat of $\text{LiBiO}_2$ .

N. Hessel Andersen, F.W. Poulsen and G. Eichinger, In: Solid State Chemistry 1982. Proceedings of the 2nd European Conference, Veldhoven, The Netherlands, 7-9 June 1982. Edited by R. Metselar, H. Heijligers and J. Schoonman (Elsevier, Amsterdam, 1983) (Studies of Inorganic Chemistry Vol. 3) 287-290.

Ac-conductivity, X-ray- and neutron powder diffraction- and DSC- experiments on solid and molten  $\text{Li}_2\text{O}-\text{Bi}_2\text{O}_3$  mixtures are reported. The ionic conductivity of  $\text{LiBiO}_2$  varies from approximately  $10^{-7}$  at  $200^\circ\text{C}$  to  $0.3 (\text{ohm.cm})^{-1}$  at  $650^\circ\text{C}$ . An abrupt increase in the conductivity occurs above  $500^\circ\text{C}$ . DSC-measurements on  $\text{LiBiO}_2$  confirm the existence of anomalous thermal properties above  $550^\circ\text{C}$ . The neutron diffraction experiments on  $\text{LiBiO}_2$  above  $550^\circ\text{C}$ , however, revealed no change in the crystal structure compared to the room temperature results. Structure refinements on the room temperature data gave results in qualitative agreement with the structure obtained from X-ray data reported in the literature.

### Neutron Diffraction Studies of Ytterbium Dihydride: The Crystal Structure at 300 K.

B. Lebech, N. Hessel Andersen, S. Stenstrup and A. Schröder Pedersen, Acta Cryst. C 39 (1983) 1475-1480.

The crystal structures of  $\text{YbD}_2$  and  $\text{YbH}_2$  have been studied by elastic neutron scattering on powdered samples at room temperature and atmospheric pressure. The neutron diffraction data have been analysed by means of the EDINP profile structure refinement program. The analyses confirmed the assumption that both compounds are isostructural with the hydrides of the alkaline-earth metals at room temperature and atmospheric pressure. All atoms are found to occupy the 4(c) position in the orthorhombic space group  $D_{2h}^{16}$  (Pnma). The atomic positions were found to be:  $x(\text{Yb}) = 0.243$ ,  $z(\text{Yb}) = 0.111$ ;  $x(\text{D}') = 0.358$ ,  $z(\text{D}') = 0.433$ ; and  $x(\text{D}'') = -0.021$ ,  $z(\text{D}'') = 0.675$ . By inference from these atomic positions, it is suggested that the room temperature high-pressure ( $p > 15$  GPa) hexagonal structure of  $\text{YbH}_2$  is  $D_{6h}^4$  ( $P6_3/\text{mmc}$ ) with the metal atoms in 2(c) positions and the hydrogen atoms in 2(a) and 2(d) positions.

Effects of Grain Boundaries and Dislocation Cell Walls on  
Void Nucleation and Growth in Aluminium During Fast Neutron  
Irradiation.

A. Horsewell, F.A. Rahman and B.N. Singh, In: Dimensional  
Stability and Mechanical Behaviour of Irradiated Metals and  
Alloys, Proceedings, Brighton, England, 11-13 April 1983,  
Vol. 1 (British Nuclear Energy Society, London, 1983)  
69-72.

High purity aluminium irradiated to fluences between  $2 \times 10^{21}$  and  $1 \times 10^{24}$  n.m<sup>-2</sup>  
(E > 1 MeV) at 120°C has been investigated by TEM. A void denuded zone is seen  
both at grain boundaries and dislocation cell walls. Enhanced void formation  
and growth occurs in a zone extending up to 10  $\mu$ m from grain boundaries in  
annealed material. In polygonized material, the presence of dislocation cell  
walls leads to cell size dependent void formation and growth; the swelling  
rate in the large cells is substantially higher than in the annealed material.

Preliminary Study of Cost Benefits Associated with Duplex Fuel  
Pellets of the LOWI Type.

J.B. Ainscough, D.N. Coucill, D.A. Howl, A. Jensen and  
I. Misfeldt, Nucl. Technol., 61 (1983) 521-532.

Duplex UO<sub>2</sub> pellets, which consist of an outer enriched annulus and a depleted  
or natural core, can provide a solution to the problem of stress corrosion  
cracking failures, which have led to constraints being placed on ramp rates  
in power reactors. An analysis of the reactor physics and the performance of  
duplex pellets is presented in the context of a 17 x 17 pressurized water re-  
actor fuel rod design. The study has been based on the particular type of  
duplex pellet in which the core and the annulus are physically separate; this  
is called "LOWI" after the Danish design. At low burnup, this fuel shows a  
significant improvement in power ramp performance compared with standard fuel.  
At higher burnup, the benefits are less certain but as the severity of the  
ramp will usually be less in high burnup fuel simply because of the reduced  
rating, the reduction in benefit may not be significant. If the gap between  
the core and annulus persists to high burnup, there will be no loss of ben-  
efit. Economic calculations and a cost-benefit analysis are presented to show  
the number of extra full-power hours of reactor operation that must be ob-  
tained in order to outweigh the additional fabrication costs associated with  
this fuel.

### The Cutting Process, Chips and Cutting Forces in Machining CFRP.

A. Koplev, Aa. Lystrup and T. Vorm, Composites 14 (1983) 371-376.

The cutting of unidirectional CFRP, perpendicular as well as parallel to the fibre orientation, is examined. Shaping experiments, 'quick-stop' experiments, and a new chip preparation technique are used for the investigation. The formation of the chips, and the quality of the machined surface is discussed. The cutting forces parallel and perpendicular to the cutting direction are measured for various parameters, and the results correlated to the formation of chips and the wear of the tool.

### Power Ramp Performance of Vipac Fuel.

P. Knudsen, C. Bagger and H. Carlsen, Res Mech. 8 (1983) 39-52.

Vibrationally compacted  $UO_2$  powder fuel ('vipac') in Zircaloy cladding may conceivably perform better than sintered pellet fuel in power ramp situations, because the smaller fuel particles may give lower stress concentrations in the cladding at hard fuel-cladding contact. It is, therefore, of interest to examine experimentally the power ramp performance of vipac fuel.

Four Zircaloy-clad vipac pins and one pellet pin were ramp-tested at a burnup of 17,900 MWD/t $UO_2$ . Three of the vipac pins failed in fast ramps at peak heat loads of 411-459 W/cm and the pellet pin failed at 348 W/cm. The fourth vipac pin was conditioned at 379 W/cm for 2 d: with a slow ramp rate of 5 W/cm min, an overpower level of 528 W/cm was reached without failure indication.

An evaluation of the local overpower levels and the eddy-current observations showed that the vipac pins had an advantage of about 80 W/cm over the pellet pin in terms of minimum heat load required for interaction and defec-tion. The many cladding cracks seen in one of the failed vipac pins had the usual brittle appearance attributed to stress-corrosion cracking.

### Experience in Fabrication Technology and Performance of Water Reactor Fuel.

P. Knudsen, H. Bairiot and S. Sandklef, In: Nuclear Power Experience, Proceedings, Vienna, 13-17 September 1982 (IAEA, Vienna, 1983) 59-81.

The major proportion of the world's operating nuclear power reactors are water-cooled and have uranium dioxide fuel clad in zirconium alloy. The prevalent reactor types are the Western PWR and BWR, the USSR WWER (PWR) and RBMK (BWR), and the Canadian CANDU (PHWR). The fuel assemblies consist of bundles of thin fuel rods; loading/reloading takes place during the scheduled shut-

downs, except for the RBMK and CANDU reactors, which are loaded during operation. There are now more than 200 water-cooled power reactors in operation with a combined net installed capacity exceeding 130,000 MW(e). As a result, large-scale industrial manufacturing capacity has been developed by the reactor vendors and some independent fuel suppliers. The fuel performance has been improved and the failure rate is now generally satisfactory. Further improvements are under way which are expected to enable a significant relaxation of existing operating restrictions while maintaining a virtually zero-failure rate, both in base-load and in load-following operation. The paper summarizes the current performance, licensing and design requirements. Developments in commercial fabrication technology and product quality are described. An overview is given of performance experience under normal operating conditions and of the observed causes of failure and their elimination.

### **The Risø Fission Gas Project.**

P. Knudsen, C. Bagger, H. Carlsen, I. Misfeldt and M. Mogensen, In: Summary Report on the OECD-NEA-CSNI/IAEA Specialists' Meeting on Water Reactor Fuel Safety and Fission Product Release in Off-Normal and Accident Conditions, Risø, 16-20 May 1983 (IAEA, Vienna, 1983) (IWGPPT/16) 318-333.

The Risø Fission Gas Project has provided experimental data on fission gas release (FGR) from high-burnup water reactor fuel. The data are well-characterized with respect to pre-irradiation measurement, irradiation and post-irradiation examination, thus enabling their use in fuel performance code validation.

The experimental data were obtained with 12 Zircaloy-clad  $\text{UO}_2$  pellet fuel pins, irradiated to burnups in the range 27,000-36,000 MWD/tU (pin average, peak pellet 43,700 MWD/tU). Most of the fuel pins were subjected to short-term reirradiations at increased power levels ("bump testing"), in order to simulate postulated power increases late in life. The 11 bump tests covered a range of bump terminal levels (BTL) of 301-434 W/cm (peak pellet), with hold time of 24 h except for one test at 72 h. The axial power shape during the bump testing differed from the base irradiation, thus each bump test was in fact a whole series of experiments with a range of BTLs.

The integral pin FGR resulting from the bump testing was in the range 0-16%, increasing with BTL above 375 W/cm (peak pellet). Owing to the form of the axial power distributions, local release data were emphasized in the project, some of the observations being:

- (a) PG and Cs-137 releases seemed to correlate with local BTL as well as axial and radial position, and to do so in a similar manner; at the highest local BTL investigated (414 W/cm), local FGR had apparently saturated at 40% within 24 h;

- (b) the local releases increased with local BTL above 350 W/cm;
- (c) bump testing to about 414 W/cm virtually emptied the central region of the fuel for PG and Cs-137;
- (d) the release measurements and the ceramographic observations for the bump test with 72 h hold time suggest that this longer time may have given more local release for the lower local BTLs.

#### **Void-Swelling in Cold-Worked Copper During HVEM Irradiation.**

**T. Leffers, B.N. Singh, S.N. Buckley and S.A. Manthorpe,**  
**J. Nucl. Mater. 118 (1983) 60-67.**

Void formation and growth in pure cold-worked copper and the parallel evolution in dislocation structure during HVEM irradiation have been investigated. Degrees of cold work in the range 10-90% rolling reduction and irradiation temperatures in the range 250-450°C have been covered. The cold-worked structure basically survives irradiation. Initially (for low degrees of cold work), void density and swelling rate increase with increasing degree of cold work; they then level off and eventually start decreasing with further cold work. The decrease in swelling rate cannot be explained in terms of a simple increase in dislocation density; one will have to consider the real, heterogeneous dislocation distribution.

#### **Additive Strengthening.**

**H. Lilholt, In: Deformation of Multi-Phase and Particle Containing Materials. Proceedings of the 4th Risø International Symposium on Metallurgy and Materials Science, Risø, 5-9 September 1983. Edited by J.B. Bilde-Sørensen, N. Hansen, A. Horsewell, T. Leffers and H. Lilholt (Risø National Laboratory, Roskilde, 1983) 381-392.**

A review is presented on the addition of strengthening contributions, with emphasis on dispersion hardened materials. Rules of addition are established for obstacles of intermixed distributions and for obstacles of regional distributions. The strength contributions are discussed: the mean stress, the "source-shortening" stress and the forest stress. Materials of non-homogeneous microstructure can behave in two different ways: unrelaxed behaviour and relaxed behaviour. The reasonably well understood microstructure and internal stresses of dispersion hardened materials are described. The differences between the stress values from an averaged dislocation arrangement and a localized arrangement are discussed.

Limsamlinger (Adhesive Bonding).

H. Lilholt, Aa. Lystrup and H. Toftegaard, Risø-M-2387  
(1983) 62 pp.

Adhesive bonding is treated in three chapters: The theory of stresses in adhesive bonds, an experimental investigation of the strength of adhesive bonds, and practical considerations for adhesive bonding. The adhesive bonding between steel and glass fibre reinforced polyester is discussed.

Fiberforstærket plast til hårdtbelastede konstruktioner (Fibre reinforced plastics for load-bearing components).

Aa. Lystrup, Risø-M-2376 (1983) 44 pp.

A survey of the use and the fabrication of advanced fibre composites is given. Many applications for space crafts, aeroplanes, cars, sports equipment, and other industrial use shown. In each case it is emphasized, which characteristics of the material have led to its use for that specific application.

A short survey of the common fabrication techniques is followed by an illustration of the fabrication of a rotor blade for a wind turbine.

The report is issued in connection with a series of slides, and contains comments to each slide. All the slides are also shown in the report.

The report and the slides are suitable to form the basis of a course on composite materials at technical and engineering schools.

The slides are available at a cost of 2000 DK-kr.

The Use of Probabilistic Fuel Performance Codes in the Planning and Evaluation of Irradiation Experiments.

I. Misfeldt, Res Mech. 7 (1983) 239-248.

In the planning of fuel irradiation experiments, as well as in their evaluation, it is important to know the influence on the test results of minor variations (tolerances) in design parameters, material properties and test specifications. This information can be obtained from a probabilistic fuel performance code such as FRP. A fuel performance code simulates detailed processes which take place during the irradiation, and its use therefore requires detailed information on both the fuel and the irradiation. The present paper describes these requirements and discusses the influence on the expected fuel performance of the most important of these specifications. Examples are given of the evaluation of actual ramp tests, especially with regard to the uncertainties expected in the test results.

## The D-COM Blind Problem on Fission Gas Release: Experimental Description and Results.

I. Misfeldt, In: Summary Report on the OECD-NEA-CSNI/IAEA Specialists' Meeting on Water Reactor Fuel Safety and Fission Product Release in Off-Normal and Accident Conditions, Risø 16-20 May 1983 (IAEA, Vienna, 1983) (IWGFPT/16) 411-422.

As part of the IAEA sponsored D-COM programme a code exercise has been conducted. The objective of the code exercise was to investigate the capability of fuel performance codes with respect to fission gas release. This report gives the experimental data, the design data, the irradiation history and the PIE data for the experiment which was used in the exercise. The experiment consisted of three minifuel pins, irradiated together to a burnup of approximately 32,000 MWd/tU at fairly low heat ratings. Two of the pins were subjected to a power transient at EOL (a bump test), the third was taken out prior to the final power transient as reference.

## Gaseous Swelling in High Burn Up Fuel During Overpower Transients.

I. Misfeldt, I.L.F. Ray and C. Baker, In: Summary Report on the OECD-NEA-CSNI/IAEA Specialists' Meeting on Water Reactor Fuel Safety and Fission Product Release in Off-Normal and Accident Conditions, Risø 16-20 May 1983 (IAEA, Vienna, 1983) (IWGFPT/16) 334-350.

In the Risø Fission Gas Project a series of slow overpower transients have been conducted in order to investigate the behaviour of high burnup water reactor fuel during a power increase.

The fuel structure was characterized in detail in both the pre-transient and post-transient conditions. The porosity developed during the transient was found to be predominant in the structure over the major part of the fuel for heat ratings above 350 W/cm. For a linear heat rating of 415 W/cm microscopic swelling started at  $r/r_0 \sim 0.8$  ( $\sim 650^\circ\text{C}$ ), and significant macroscopic swelling was observed from  $r/r_0 \sim 0.7$  ( $\sim 800^\circ\text{C}$ ).

In order to compare the observed swelling rates with published data on gaseous swelling, the fuel temperature during the transients has been estimated. The details of the temperature calculation are given in the paper. The swelling that developed during the 24-hour bump test was around 5% at  $850^\circ\text{C}$ , corresponding to swelling rates as high as 1500%/FIMA, which is several orders of magnitude higher than the swelling rates quoted in the literature for  $\text{UO}_2$  fuel around  $1500^\circ\text{C}$ .



#### Determination of Fission Gas Yields from Isotope Ratios.

M. Mogensen, Int. J. Mass Spectrom. Ion Phys. 48 (1983) 389-391.

This paper describes a method of calculating the actual fission yield of Kr and Xe in nuclear fuel including the effect of neutron capture reactions and decay. The bases for this calculation are the cumulative yields (Ref. 1) of Kr and Xe isotopes (or pairs of isotopes) which are unaffected by neutron capture reactions, and measured Kr and Xe isotope ratios. Also the burnup contribution from the different fissile heavy isotopes must be known in order to get accurate fission gas yields.

#### The Passivation of Lithium in Thionyl Chloride Non-aqueous Electrolyte.

M. Mogensen, In: Proceedings of the 9th Scandinavian Corrosion Congress, Copenhagen, 12-14 September 1983. Vol. 2. (Korrosionscentralen, ATV, Glostrup, 1983) 699-710.

The Li/SOCl<sub>2</sub> nonaqueous electrolyte system has been studied extensively with regard to its suitability as an anode-electrolyte system for high energy density battery applications. The primary difficulty associated with the use of the system is that lithium metal is not thermodynamically stable in SOCl<sub>2</sub>. It is apparently stable only because of the spontaneous formation of a thin passivating layer of LiCl.

As long as the LiCl-layer is thin the Li/SOCl<sub>2</sub> half cell performs extremely well. Because of corrosion processes, however, the LiCl-layer thickens, and the electrical resistance of the electrode increases. During some months this resistance reaches a level which is unacceptable in many battery applications.

The paper gives first an overview of the phenomenon mainly based on the literature. Next a method of monitoring the growth of the LiCl-layer by means of small amplitude ac response measurements is described, and finally a mechanistic interpretation of the measured impedance spectra is attempted.

#### Local Fission Gas Release From High Burnup Water Reactor Fuel Under Transient Conditions.

M. Mogensen and C.T. Walker, In: Summary Report on the OECD-NEA-CSNI/IAEA Specialists' Meeting on Water Reactor Fuel Safety and Fission Product Release in Off-Normal and Accident Conditions, Risø, 16-20 May 1983 (IAEA, Vienna, 1983) (IWGFPT/16) 351-358.

The paper presents results for local gas release, produced by power transients (bump tests) at the end of life of a water reactor fuel. The burnup was from 2.7-4.5% FIMA (25,000-41,000 MWd/tU). The local linear power at the bump terminal level (BTL) of the fuel examined ranged from 300-415 W/cm. The hold time at BTL was either 24 or 72 h. Around 410-415 W/cm, the local gas releases measured on pellet sized samples were 35-40%. Radial xenon release profiles measured by electron microprobe analysis showed that onset of release occurred at about 700°C. Above 1100°C, a constant release of about 95% was found.

**Mass Spectrometric Measurement of Fission Gas from Nuclear Fuel.**  
**E. Larsen, H. Egsgård and M. Mogensen, Int. J. Mass Spectrom. Ion Phys. 48 (1983) 385-388.**

Isotropic and quantitative measurements of the fission gases Xe and Kr in fuel pins are presented. The gas extracted by puncturing a fuel pin and the retained gas released by oxidizing fuel pellet size samples are compared with the calculated amount of fission gas generated.

**Corrosion of Steel in Concentrated Hydrogen Carbonate Solution.**  
**T. S. Nielsen, In: Proceedings of the 9th Scandinavian Corrosion Congress, Copenhagen, 12-14 September 1983.**  
**Vol. 2. (Korrosionscentralen, ATV, Glostrup, 1983) 607-618.**

The corrosion properties of steel in a 12% NaHCO<sub>3</sub> + 2% Na<sub>2</sub>CO<sub>3</sub> solution has been investigated with a view to application in a uranium extraction process.

Electrochemical investigations at temperatures up to 80°C revealed no major differences between pure iron, unalloyed, and a low-alloy steel. The oxidizing conditions of the process passivate the surfaces, and in the concentrated hydrogen carbonate solution pitting occurs only with large chloride concentration and low temperature.

The passive state pertains to 250°C under static conditions, and there is no erosion effect from a flowing ore slurry during the initial build-up of the oxide layer. It was unfortunately not possible to obtain so long exposure to flowing slurry, that the fully passive state could be investigated. At 300°C under static conditions, a more or less constant corrosion rate was experienced.

No incidences of stress corrosion cracking has been observed with 1000 hrs exposure.

## Thermoelasticity and Plasticity of Composites - I. Mean Field Theory.

O.B. Pedersen, Acta Metall. 31 (1983) 1795-1808.

A simple mean field model leads to a new formulation of the theory of bounds for thermoelastic composites whose phase volume fractions are known, only. In this formulation the theory expresses the equivalent transformation strain as the sum of a term proportional to the external stress and a term proportional to the inelastic strain misfit. Comparisons with selected experiments show that a realistic description of the plasticity of composites in general has to account for the effects of matrix constraint and for the effects of elastic heterogeneity. The new formulation may be combined with a dislocation theory of constrained flow. Implications for the cyclic plasticity of composites are pointed out.

## The Strength of Heterogeneous Materials - Continuum Models and Discrete Models.

O.B. Pedersen and L.M. Brown, In: Deformation of Multi-Phase and Particle Containing Materials. Proceedings of the 4th Risø International Symposium on Metallurgy and Materials Science, Risø, 5-9 September 1983. Edited by J.B. Bilde-Sørensen, N. Hansen, A. Horsewell, T. Leffers and H. Lilholt (Risø National Laboratory, Roskilde, 1983) 83-102.

The low temperature strength of heterogeneous materials is discussed in terms of combinations of composite theory, continuum plasticity and dislocation mechanics. Mean field models based on energy and stress calculations are considered for two-phase materials with parallel randomly arranged ellipsoidal inclusions of identical shapes. A short review is given of two fundamentally different models for the plasticity of particle containing metals: The continuum model and the interaction hardening model. A simple combination of elements from the two models accounts semi-quantitatively for experimental data on the workhardening, Bauschinger effect and slip line spacing of dispersion strengthened metals. Similar types of approach to the modelling of a much wider range of materials and deformation modes are considered.

## On The Surface Reaction of Hydrogen with FeTi.

A. Schrøder Pedersen, P. Juul Møller and O. Toft Sørensen, Phys. Scr. T4 (1983) 83-85.

The surface reaction of hydrogen (and of deuterium) with activated FeTi was studied by UHV volumetric vapour pressure measurements in the  $3 \times 10^{-8}$  Pa to  $3 \times 10^{-5}$  Pa range at 78 K and 88 K.

At the low temperatures a dissociative non-activated Temkin chemisorption process was found. an isosteric heat of adsorption,  $q$ , was described by the expression  $q = 24 (1 - \theta) \text{ kJ/(mole H}_2\text{)}$  for a broad range of coverages. It was demonstrated that the dissociation occurred on a non-oxidized Ti-surface and not - as has been suggested - on a segregated subsurface Fe-layer.

#### Magnesium for Hydrogen Storage.

A. Schrøder Pedersen, J. Kjøller, B. Larsen and B. Vigeholm,  
Int. J. Hydrogen Energy 8 (1983) 205-211.

A study of the hydrogenation characteristics of fine magnesium powder during repeated cycling has been performed using a high-pressure microbalance facility. No effect was found from the cycling regarding kinetics and storage capacity. The reaction rate of the absorption process was fast at temperatures around 600 K and above, but the reversed reaction showed somewhat slower kinetics around 600 K. At higher temperatures the opposite was found. The enthalpy and entropy change by the hydrogenation, derived from pressure-concentration isotherms, agree fairly well with those reported earlier.

#### A Kinetic and Thermodynamic Study of the Reaction of Hydrogen and Deuterium with FeTi at Low Pressures.

A. Schrøder Pedersen, P.J. Møller and O. Toft Sørensen,  
Ber. Bunsen-Ges. Phys. Chem. 87 (1983) 104-112.

The initial reaction of hydrogen and deuterium with clean surfaces of FeTi powder, following a five-cycles activation procedure using hydrogen, were studied by volumetric and microgravimetric techniques. - It was concluded that within the 78 K to 88 K temperature range a non-activated dissociative Temkin chemisorption process occurred upon a Ti surface layer, while at temperatures within the 300 K to 500 K range a dissolution process took place limited by diffusion in the  $\alpha$ -phase.

#### Properties of LiI-Alumina Composite Electrolytes.

F.W. Poulsen, N. Hessel Andersen, B. Kindl and J. Schoonman,  
Solid State Ionics 9/10 (1983) 119-122.

LiI and 14 different LiI-high surface area  $\text{Al}_2\text{O}_3$  composite electrolyte materials have been examined by the ac-admittance method. The presented data span the temperature range from 25°C to 300°C, and the composition range from

20 to 70 mole %  $\text{Al}_2\text{O}_3$ , respectively. Maxima are observed in the isothermal conductivity versus composition plots at 40-50 mole %  $\text{Al}_2\text{O}_3$ . At  $25^\circ\text{C}$  the best material has a specific conductivity equal to  $3.75 \cdot 10^{-5}$  S/cm, or a factor 100 to 1000 higher than for LiI at the same temperature. The enhanced conductivity results from an increase in the preexponential factors and a minimum in the overall activation energy for conduction occurring between 40 and 50 mole % alumina. The compositional dependence of the conductivity is discussed on basis of a pore-model. Preliminary analysis of Hebb-Wagner polarization data for the cell Li/LiI/C indicates that electronic leakage currents in LiI are carried by electrons.

#### NMR Studies of Lithium Iodide based Solid Electrolytes.

R. Dupree, J.R. Howells, A. Hooper and F.W. Poulsen,  
Solid State Ionics 9/10 (1983) 131-134.

In mixtures of LiI with  $\gamma\text{Al}_2\text{O}_3$  the ionic conductivity is found to increase by up to three orders of magnitude over pure LiI. NMR measurements of  $^7\text{Li}$  relaxation times were performed on both anhydrous LiI and a mixture of LiI with 30m/o  $\gamma\text{Al}_2\text{O}_3$ . The relaxation is found to be purely dipolar in origin for the pure halide with correlation times consistent with conductivity data. However two relaxation rates are found in the composite material which indicate that ~50% of the  $^7\text{Li}$  ions are in a radically different environment.

#### Role of Inert and Reactive Gases in Void Nucleation.

A.J.E. Foreman and B.N. Singh, In: Dimensional Stability and Mechanical Behaviour of Irradiated Metals and Alloys, Proceedings, Brighton, England, 11-13 April 1983, Vol. 1 (British Nuclear Energy Society, London, 1983) 95-99.

Gas seems to be an essential pre-requisite for void nucleation, since vacancy clusters collapse into loops without gas stabilization. The inert gases are very effective, but many quenching and electron irradiation experiments have demonstrated that the residual gases in materials can readily nucleate voids. Furthermore, low dose neutron irradiations of pure metals show that reactive gases nucleate voids very rapidly. However a second population can form if the helium production rate is high. The mechanisms and experimental evidence for nucleation by inert and reactive gas atoms are discussed, with special emphasis on the temperature dependent void densities produced by electron irradiations.

**Void Formation in Pure Aluminium Irradiated with High Energy Electrons and Gamma Quanta (in Russian).**

V.V. Gann, L.S. Ozhigov, V.A. Yamnitskij, B.N. Singh, T. Leffers, and J.B. Bilde-Sørensen, Voprosy Atomnoi Nauki i Tekhniki, Series Fizika Radiatsionnykh Povrezhdenii i Radiatsionnoye Materialovedenie 24 No. 1 (1983) 41-46.

The spatial distribution of displaced atoms and helium atoms and the damage energy spectrum of the primary displaced atoms are calculated for thick aluminium specimens irradiated with 225 MeV electrons. The void and dislocation structure of irradiated Al specimens were studied by transmission electron microscopy.

**Effects of Cold-Work on Void Nucleation and Growth in an Austenitic Stainless Steel during Heavy-Ion- and Electron Irradiation.**

V.N. Voevodin, B.V. Matvienko, B.N. Singh and T. Leffers, In: Dimensional Stability and Mechanical Behaviour of Irradiated Metals and Alloys, Proceedings, Brighton, England, 11-13 April 1983, Vol. 1 (British Nuclear Energy Society, London, 1983) 33-36.

Specimens of an austenitic stainless steel in solution-treated and cold-worked condition were irradiated with 1 MeV Cr ions in an accelerator and with 1 MeV electrons in a high voltage electron microscope. The evolution in dislocation and void structure is followed for each type of specimen and each type of irradiation, and the results are compared.

**Continuum Mechanics Characterisation of Damage in Composite Materials.**

R. Talreja, DCAMM Report No. 268 (1983) 40 pp.

The mechanical response of a composite material damaged in fatigue, or in any other loading mode, is characterized by means of a set of vector fields, each representing a damage mode. Constitutive equations are derived for isothermal small-deformation behavior following attainment of a damage state. For small damage, the elastic constants of a damage state are related to those of the undamaged state. These relations provide useful means for characterizing the development of damage, for instance during fatigue.

**Stepwise Isothermal Analysis. A New Technique in Thermo-gravimetry and Dilatometry.**

O. Toft Sørensen, In: 5. Convegno Nazionale di Calorimetria ed Analisi Termica. Atti del Convegno. Trieste, 14-16 December 1983. Edited by A. Cesàro and S. Memari (Universita Degli Studi di Trieste, Trieste, 1983) 25-39.

Stepwise Isothermal Analysis is a new technique which has proved very useful in thermogravimetric and dilatometric studies. Among the advantages can be mentioned: Improved resolution of close-lying reactions; Reactions take place at near-equilibrium conditions; Accurate kinetic data can be obtained; Data for all relevant reactions can be obtained in a single run. In the paper several examples demonstrating these advantages in thermal decomposition studies as well as in dilatometric sintering studies are discussed.

**Cycling Performance of the Magnesium - Magnesium Hydride System.**

B. Vigeholm, J. Kjøller, B. Larsen and A. Schrøder Pedersen, In: Hydrogen as an Energy Carrier, Proceedings of the 3rd International Seminar. Lyon, 25-27 May 1983. Edited by G. Imarisio and A.S. Strub (D. Reidel Publication Company, Dordrecht, 1983) 442-456.

Some of the magnesium properties essential to the applicability of the reaction  $Mg + H_2 \rightleftharpoons MgH_2$  as a hydrogen storage system have been investigated. Magnesium powders with particle size smaller than 50  $\mu m$  average diameter were cycled, over 30 to 500 cycles, at 675 K (400°C) and at adsorption pressures, 2 - 3 MPa.

The powders adsorbed and desorbed 60 - 95% of the theoretically maximal amount of hydrogen given by stoichiometric  $MgH_2$ . The reaction rates changed negligibly over the extent of the experiments permitting the mentioned hydrogen exchange to be completed in less than 20 min. At the experimental conditions absorption and desorption rates were very similar, the desorption generally slightly the faster, 4 - 9 min. compared to 7 - 10 min. The storage capacity dropped to approximately 60% in the experiments performed at constant, high temperature. This is assigned partly to magnesium oxide formation partly to sintering of the particles into larger conglomerations.

**Hydrogen Sorption Performance of Pure Magnesium During Continued Cycling.**

B. Vigeholm, J. Kjøller, B. Larsen and A. Schrøder Pedersen, Int. J. Hydrogen Energy 8 (1983) 809-817.

Preliminary investigations of the hydrogen absorption-desorption by commercially pure magnesium powder under continuous operation show little or no reduction in hydrogen capacity up to 70 cycles and high temperature exposure exceeding 1200 h. Absorption was studied at 260°C-425°C and hydrogen pressures up to 2.0 MPa above equilibrium. Desorption was with a few exceptions done at 400°C at hydrogen pressures below 150 kPa. For practical application the hydrogen exchange may be limited to 75-90% of the complete metal to stoichiometric hydride reaction. A change of the macroscopic structure of the powder into a highly porous, sintered agglomerate did not reduce the hydrogen capacity or the reaction rate. Although this change in structure caused no deterioration of the cycling performance a further development may not be acceptable. For observation over a much larger number of cyclings a fully automated, triple line cycling facility permitting simultaneous testing under different conditions has been constructed.

#### Formation and Decomposition of Magnesium Hydride.

B. Vigeholm, J. Kjøller, B. Larsen and A. Schrøder Pedersen,  
J. Less-Common Metals 89 (1983) 135-144.

The absorption of hydrogen in magnesium (purity, 99.8%-99.94%) was studied in the temperature range 260-425°C and at pressures from the equilibrium value to 2 MPa above equilibrium. At constant temperature the absorption rate depends on the pressure whereas total absorption is attained in approximately the same time regardless of the pressure. The final composition is very close to stoichiometric  $MgH_2$  except at the lowest temperatures and highest pressures investigated when the reaction becomes extremely slow or ceases completely at 80%-90% of the stoichiometric composition. Accurate determination of the relative molar enthalpy gives a value of  $-70 \text{ kJ (mol } H_2)^{-1}$ . No hysteresis in the ordinary sense was observed, but although no change in the plateau pressure occurred desorption of from 5%-15% of the remaining hydrogen required almost zero pressure. The actual value appears to be dependent on the material.



## LECTURES

### Recovery of a Creep-Induced Dislocation Network.

J.B. Bilde-Sørensen, presented at a Discussion Meeting on "Significance of Internal and Threshold Stresses to Creep Deformation of Engineering Alloys", The Metals Society, London, 18 May 1983. (Not available).

The annealing of a creep-induced dislocation network has commonly been described by the Friedel model or similar models which predict that the change in average length per time unit is inversely proportional to the average length. However, experimental measurements of the annealing after a stress removal shows an initial phase of fast recovery after which the recovery rate rapidly decreases to a value close to zero. Furthermore, annealing curves for the same material with different initial dislocation density exhibit recovery rates which differ vastly at identical current dislocation density (J.Hausselt and W.Blum, Acta Metall. 24 (1976) 1027-1039).

The present paper suggests a dislocation model for recovery in which sites for strong recovery are created by the glide process. In this model the recovery rate therefore depends not only on dislocation density but also on the strain history, which explains how networks with the same total density can exhibit different recovery rates. After a stress removal no new sites for strong recovery are created, and as the sites created before the stress removal are exhausted, the recovery rate will approach zero in agreement with the experimental findings.

This model suggests that a friction stress measured by the method of cumulative incubation periods contains a component from the dislocation network left in the material when the recovery sites have been exhausted. In a pure single-phase material this component is probably the major contribution. At a first sight the success of the friction stress approach may therefore seem surprising. However, the difference between the initial and the final dislocation density (and thereby between the applied stress and the measured friction stress) depends on the number and the strength of the recovery sites at the time of the stress removal, and is therefore in some way related to the recovery rate during steady-state deformation. The good correlation obtained by the friction stress approach may therefore not be quite fortuitous.

### Characteristics of Brazed Joints in High Temperature Materials.

G.E. Sheward and J. Christensen, presented at the 4th International Conference held by the British Association for Brazing and Soldering, London, 1-3 November 1983. (Available as UKAEA Report No. DCO 7563(S)).

Brazed joints are being increasingly used to manufacture assemblies for high temperature applications - in many cases for use in critical situations in which the penalty of failure is high. Although much information has been published on the properties of brazed joints, the data is frequently related to a specific application, and so there is a shortage of general information on the basic properties of brazed joints made in high temperature materials. This paper will therefore review their characteristics, discussing the effects of the brazing cycle, the parent metal/filler metal reactions, component and joint design and heat treatment on the properties of the brazed component. The application of quality control procedures can be applied with confidence and make the high temperature brazing process one of the most reproducible of all the joining processes. Finally, recommendations are made about further research and development to obtain a better knowledge of the basic nature of brazed joints and the brazing process, so that it can be more widely applied with greater confidence.

**Brudmekanisk Bestemmelse af Duktil Revneudbredelse (Determination of Ductile Crack Growth using Fracture Mechanics).**

C.P. Debel, presented at the Experimentel Mekanik Dag 1983, Risø, 26 Oktober 1983. (Not available)

The single-specimen procedure adopted at Risø National Laboratory in measuring the J-integral based fracture resistance curve is presented. The method is based on applying the direct current, potential difference technique (DC-PD) in measuring the amount of ductile crack growth obtained.

**Physical Influence on the Masterslave Manipulator as a Result of the Working Conditions.**

H. Hougaard, presented to the Euratom Working Group on Hot Cells and Remote Handling, Petten, Holland, 8-10 June 1983. (Transcript available).

In the hot laboratories much of the work is performed remotely by the aid of master-slave manipulators. As the handling often is done behind thick walls and therefore with a large distance between the operator and the object, the handling is much more difficult and heavy than under direct circumstances. These conditions may lead to different disabilities for the operators.

The results of a detailed analysis of the factors which may affect the operation performed at the hot cells at Risø National Laboratory are described in this paper. These results are supplemented with experience from other hot laboratories within the EEC countries.

## **Plastisk Deformation af Metaller (Plastic Deformation of Metals).**

**T. Leffers**, presented to Dansk Metallurgisk Selskab, Lyngby, 8 March 1983. (Not available).

The present stage of our understanding of the plastic deformation of metals is described - with special emphasis on the relation between the models for plastic deformation derived from the microstructure and those derived on the basis of continuum mechanics.

## **Plast, Fibre og Møllevinger (Plast, Fibres and Blades for Windturbines).**

**Aa. Lystrup**, presented at Åbent Hus på Risø, Risø, 4-5 June 1983. (Not available).

After a short introduction to fibre reinforced plastics, a fabrication method for blades for large wind turbines is given.

## **Mechanical Behaviour and Fission Product Release in Overpower Transients at Extended Burnup.**

**I. Misfeldt**, presented at the IAEA Specialists' Meeting on Pellet-Cladding Interaction in Water Reactor Fuel, Seattle, Washington, 3-5 October 1983 (Proceedings to be published as an IAEA report).

An important factor for the utilization of water reactor fuel at extended burnup is the PCI behaviour during operational transients.

The data from the internationally sponsored Risø Fission Gas Project have provided significant information regarding the fission product release and swelling of  $\text{UO}_2\text{-Zr}$  fuel pins with burnup in the range of 38,000-44,000 MWD/tU, peak pellet, during mild overpower transients (bump tests).

The pellet cladding mechanical interaction during the transient tests was large enough to cause permanent deformation down to local heat ratings around 300 W/cm. The mechanical interaction was promoted by very rapid gaseous swelling, which developed during the transients. The local swelling increase during the short transient tests was measured by QIA and found to be as high as 8% at local, calculated temperatures of 800°C.

The local release of the volatile fission products Cs and I was measured by radial gamma scanning and found to be very similar to the measured release of noble fission gas. Radial EMPA measurements showed that the Xe release started at local, calculated temperatures of about 700°C. Above 1100°C a release of about 95% was found.

Although significant mechanical interaction as well as release of volatile fission products were observed, only one of the twelve tests resulted in cladding failure. This is attributed to the slow approach to the full test power levels, where a power increase rate of about 5 W/cm/h (peak pellet) was generally used.

## Summary of the Code Results for the D-COM Blind Problem.

I. Misfeldt, presented at the OECD-NEA-CSNI/IAEA Specialists' Meeting on Water Reactor Fuel Safety and Fission Product Release in Off-Normal and Accident Conditions, Risø, 16-20 May 1983. (Not available).

As part of the IAEA sponsored D-COM programme, a code exercise is conducted with the objective of investigating the capability of fuel performance codes with respect to fission gas release. A total of 15 codes from 12 countries have submitted code results prior to the disclosure of the experimental results. The code results are presented in summary form and compared with the obtained experimental results. The exercise shows some deviations between the codes in the temperature predictions for the experiment as well as difficulties in the calculations of the experimentally observed high gas release during the short bump test at end-of-life.

## Utilization of the Isotopic Composition of Xe and Kr in Fission Gas Release Research.

M. Mogensen, presented at the Enlarged Halden Programme Group Meeting, Loen, Norway, 23-28 May 1983. (Transcript available).

Two examples of how the measured fission gas isotopic composition can be used in the study of fission gas release phenomena are given. In the first example the ratios of Kr85/Kr86 in released and retained gas are used for calculation of the "average time" when the gas was released. This "average time" may be used in code qualification. In the second example the degree of conversion of unstable Xe135 to stable Xe136 is derived from the measured ratio of Xe136 to Xe131+Xe132. This conversion is of importance in the calculation of the total Xe generation during irradiation.

## Dislocation Microstructures in Fatigued Metals.

O.B. Pedersen, Invited lecture at the Annual Meeting of the Scandinavian Society for Electron Microscopy, Trondheim, 5-8 June 1983 (The Norwegian Institute of Technology, Trondheim, 1983).

Recent studies of metal fatigue have concentrated on the dislocation microstructures built up in ductile single crystals and polycrystals in the early stages of plastic strain controlled fatigue. The structures are heterogeneous, consisting of different types of clumps of densely packed dislocations embedded in relatively undislocated crystal. Theory and experiments suggest that the clumps are partly elastically accommodated: they suffer

plastic flow at large amplitudes but not normally at small amplitudes. The size of the clumps is of the order of microns, and simple low-magnification TEM is used to characterise them. Cross-slip of screw dislocations plays a decisive role in the evolution of these structures.

At present a fairly detailed picture is emerging of the successive events in the fatigue process, especially for metals with high stacking fault energies. This will be illustrated using TEM observations correlated by fatigue diagrams, plots of cycle number versus plastic strain amplitude. The stages of unidirectional hardening and ductile fracture are represented in these diagrams at a quarter-cycle. Each mechanism of cyclic hardening and fatigue cracking is mapped on a corresponding field bounded by scatter bands.

#### **Thin Film Lithium Aluminium Negative Plate Material.**

**J.R. Owen, W.C. Maskell, B.C.H. Steele, T.S. Nielsen and O. Toft Sørensen, presented at the 4th International Conference on Solid State Ionics, Grenoble, France, 4-8 July 1983. (To be published in Solid State Ionics).**

LiAl thin film electrodes were formed by cathodic conversion of the surface of a 50 micron Al foil in a propylene carbonate based electrolyte. The films were cycled both in Li-doped poly(ethylene oxide) and propylene carbonate. On charging, an overpotential of 40 mV was required to sustain growth of the LiAl phase, but the diffusional overpotential was minimal. A simple diffusional model for the discharge predicted serious rate limitation due to slow diffusion in the  $\alpha$  phase. However, measurements of the diffusion coefficient gave values many orders higher than previously reported and even greater values were required to explain the discharge rates. Cycling behaviour showed that the electrode degradation was less severe in the polymeric electrolyte than in propylene carbonate.

#### **Corrosion Aspects at High Level Waste Disposal in Salt Domes.**

**K. Rørbo, presented at the 12ème Réunion du Groupe de Travail "Corrosion Nucléaire" de la Fédération Européenne de la Corrosion, Risø, 30 September 1983. (Transcript available).**

In the Elsam/Elkraft waste management project it is planned that the high-level waste is glassified, encapsulated in canisters and finally deposited in a deep hole drilled in a salt dome. In the present report corrosion aspects of the canisters after deposition are discussed. The chemical environment will probably be a limited amount of brine coming from brine inclusions in the surrounding salt and moving up against the temperature gradient, the temperature at the canister surface being in the range of 100-150°C. The possible types of corrosion and the expected corrosion rates for a number of po-

tential canister materials (mild steel, austenitic and ferritic stainless steels, Ni-base alloys, copper, titanium and a few combinations of materials) are discussed. Mild steel (possibly combined with an inner layer of copper or titanium) might possibly be an appropriate choice of material for the canister.

#### Grain Boundary Related Effects in Aluminium During 600 MeV Proton Irradiation at Different Temperatures.

B.N. Singh, T. Leffers, W.V. Green and M. Victoria, presented at the 3rd Topical Meeting on Fusion Reactor Materials, Albuquerque, New Mexico, 19-22 September 1983. (To be published in J. Nucl. Mater.).

Samples of high-purity aluminium were irradiated with 600 MeV protons at temperatures in the range 130 to 433°C; in these experiments, 615 and 125 appm of hydrogen and helium, respectively, are produced per dpa. Bubble formation and growth at grain boundaries and in the zone adjacent to the bubble-denuded zone are described. Precipitation at grain boundaries and migration of grain boundaries during irradiation are also reported.

#### Cavity Formation in Aluminium Irradiated with a Pulsating Beam of 225 MeV Electrons.

B.N. Singh, J.B. Bilde-Sørensen, T. Leffers, L.S. Ozhigov and V.V. Gann, presented at the 3rd Topical Meeting on Fusion Reactor Materials, Albuquerque, New Mexico, 19-22 September 1983. (To be published in J. Nucl. Mater.).

High-purity aluminium was irradiated with a pulsating beam of 225 MeV electrons to a maximum dose of 0.035 dpa with a helium generation rate of up to 17 appm/dpa. The irradiated samples contained cavities in the size range 5 to 70 nm. In a zone of up to 14  $\mu$ m from the grain boundaries cavity size and density are found to be greater than in the grain interior.

#### Temperature Dependence of Void and Bubble Formation and Growth in Aluminium During 600 MeV Proton Irradiation.

M. Victoria, W.V. Green, B.N. Singh and T. Leffers, presented at the 3rd Topical Meeting on Fusion Reactor Materials, Albuquerque, New Mexico, 19-22 September 1983. (To be published in J. Nucl. Mater.).

As a part of a continuing program, we report in the present paper results obtained from irradiating pure aluminium samples in the PIREX facility installed in the 600 MeV proton beam of the accelerator at the Swiss Institute for Nuclear Research (SIN).

The aluminium foils have been irradiated at 8 different temperatures in the range from 130° - 430°C, to displacement doses of up to 5 dpa and helium contents of over 1000 appm.

The TEM examinations have shown that at all irradiation temperatures and displacement doses, helium bubbles are formed uniformly through the whole grain interior. No voids are observed at temperatures above 160°C.

At all temperatures, irradiation induced dislocations have been observed, most of them linked to bubbles. At higher temperatures and doses, clear evidence of irradiation induced precipitation has been observed; the precipitates are normally decorated with helium bubbles.

### Relative Role of Gas Generation and Displacement Rates in Cavity Nucleation and Growth.

B.N. Singh and A.J.E. Foreman, presented at the 3rd Topical Meeting on Fusion Reactor Materials, Albuquerque, New Mexico, 19-22 September 1983. (To be published in J. Nucl. Mater.).

Problems of helium diffusion and clustering during irradiation are analysed. Using the "homogeneous" nucleation theory, the effect of damage rate on cavity density is calculated for different gas generation to damage rate ratios. The influence of gas mobility on cavity nucleation has been evaluated. The calculations have been carried out for aluminium; the results are compared with the experimental results on high-purity aluminium irradiated with 600 MeV protons.

### The Problem of Simulation.

B.N. Singh and T. Leffers, presented at a Workshop on Evaluation of Simulation Techniques for Radiation Damage in the Bulk of Fusion First Wall Materials, Interlaken, Schweiz, 27-30 June 1983. (Not available).

The present situation for the simulation of the behaviour of fusion first wall materials is described. It is underlined that ideal simulation is not possible at the present stage, because the physical conditions in all available simulation facilities differ from those in the real first wall, and because we do not know the scaling laws to apply to go from model experiment to first wall conditions. In this situation we have to use the existing simulation facilities in an effort to understand the individual processes that combine to produce the damage in the first wall - and hence come to know the scaling laws.

## Effects of Impurities, Gases and Thermomechanical Treatments on Cavity Nucleation and Growth.

B.N. Singh, presented at Hanford Engineering Development Laboratory, Richland, Washington, 3 October 1983. (Not available).

First of all, a general description will be given of the activities in the field of radiation damage studies at Risø National Laboratory. Effects of under- and over-size impurity atoms on void nucleation and growth will be considered. It will be shown that the impurity effects can be maintained even in the presence of pre-implanted gas atoms. The problem of structural evolution during irradiation in heterogeneous and segregated forms will be considered. The influence of mechanically or thermomechanically induced dislocation structure on nucleation or growth of voids during irradiation will also be discussed.

## Dilatometric Sintering Studies on Ceria-Zirconia Powders.

M. El Sayed Ali, O. Toft Sørensen and S. Meriani, presented at a Conference on the Science of Ceramics, Saint-Vincent, Italy, 27-30 June 1983. (Proceedings to be published).

The sintering behaviour of Ceria-Zirconia powders of metallorganic origin is in the present work compared to that of ground and mixed pure crystalline oxides, with a new Stepwise Isothermal Dilatometry (SID) method which has the advantage that both mechanism and activation energy can be determined in a single experiment. For the mixed oxides the controlling mechanism was determined to be grain boundary diffusion of the cations. Due to the poor sinterability no kinetic analysis could be performed on powders of metallorganic origin.

## Aspects of Fatigue in Composite Materials.

R. Talreja, presented at the Gordon Research Conference on Composite Materials, Santa Barbara, California, 17-21 January 1983; at the Aeronautical Research Institute of Sweden, Bromma, 23 February 1983; at the Royal Institute of Technology, Stockholm, 24 February 1983; at the Center for Composite Materials and Structures, Blacksburg, Virginia, 19 May 1983; at The Center for Adhesion Science, Blacksburg, Virginia, 26 May 1983; at Owens - Corning Fiber Glas Technical Center, Granville, Ohio, 13 June 1983; at The Materials Research Laboratory,



Washington University, St. Louis, Missouri, 17 June 1983;  
at a Conference on the Review of Quantitative Non-Destructive  
Evaluation, Santa Cruz, California, 7-12 August 1983;  
at the U.S.-Swedish Workshop on Fatigue and Fracture of  
Graphite Epoxy Composites, Stockholm, 8-9 December 1983.  
(Not available).

The characterisation and modelling of fatigue damage in polymer matrix fibre  
composites were discussed.

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